

Predictive Equipment Maintenance



Oil Analysis Handbook

Third Edition

Preface

Welcome to the third edition of the In-service Oil Analysis Handbook.

It has been a few years since the publication of the first edition of Spectro Scientific's In-Service Oil Analysis Handbook. Our original goal was to compile a comprehensive reference book of common in-service oil analysis techniques to help readers understand and choose the right technique and instrumentation for their needs. We had to limit the scope in the first two editions because of the amount of effort needed to cover all the topics.

In-service oil analysis for condition based maintenance covers a wide array of topics. I am pleased to say that in this third edition, we are much closer to our goal. We reorganized the structure of the content for better clarity and we added articles to cover more topics and instruments associated with oil analysis. Also, we rewrote several articles including the latest developments on the market. As we learned more from our customers about their successes using on site oil analysis, we developed case studies that you can find in this edition.

This work is not possible without the time and effort from the contributing authors: Patrick Henning, Daniel Walsh, Robert Yurko, Ken Caldwell, Thomas Barraclough, Maria Bartus, Randi Price, John Morgan, Aifeng Shi and Yuegang Zhao from Spectro Scientific and Ray Garvey from Emerson Process Management. Special thanks to Pat Henning, Chief Technology Officer of Spectro Scientific for updating the entire technology section and to Sandra Schiller for managing the project.

In-service oil analysis technologies have advanced in the past few years. Innovations in new products have brought on-site oil analysis into the hands of reliability professionals and mechanics. Simple analytical tools enable engineers to make maintenance decisions quicker and with more confidence. We are so pleased to share the recent developments in this field and look forward to more progress down the road.

Yuegang Zhao

Contents

Preface	3
---------------	---

Section 1: Introduction

Chapter 1: In-service Oil Analysis for Condition Based Maintenance	7
--	---

Chapter 2: Oil Sampling Best Practices	14
--	----

Section 2: Technologies

Chapter 3: Elemental Analysis	23
-------------------------------------	----

3.1: Rotating Disc Electrode OES (RDE-OES)	24
--	----

3.2: Elemental Analysis: FPQ-XRF for Large Particles	31
--	----

Chapter 4: Particle Analysis	37
------------------------------------	----

4.1: Dynamic Equilibrium – Large Wear Particles in Oil Lubricated Systems	37
---	----

4.2: Ferrography – Particle Size and Morphology Analysis	39
--	----

4.3: Particle Count	41
---------------------------	----

4.4: Ferrous Particle Analysis	43
--------------------------------------	----

4.5: LaserNet Fines® – Particle Count, Particle Shape Analysis and Ferrous Particle Analysis ..	44
---	----

Chapter 5: Infrared Spectroscopy	52
--	----

5.1: Direct Infrared Spectroscopy Based on Grating Optics	53
---	----

5.1.1: FluidScan Introduction	53
-------------------------------------	----

5.1.2: TAN-TBN Measurement Using Infrared Technique	57
---	----

5.1.3: Water Measurement Using the Infrared Technique	64
---	----

5.1.3.1: Dissolved Water Measurement	64
--	----

5.1.3.2: Free Water and Total Water Measurement	66
---	----

5.2: Filter Based Infrared Techniques	70
---	----

Chapter 6: Viscosity	72
----------------------------	----

6.1: Laboratory Kinematic Viscometers	72
---	----

6.2: Solvent-free Portable Viscometer	74
---	----

Chapter 7: Fuel Dilution in Engine Oil	79
--	----

Section 3: Oil Analysis Practices and Success Stories

Chapter 8: Lubricant Mix Up – How to Catch it with Oil Analysis	85
Chapter 9: Route-Based Oil Analysis	88
Chapter 10: Oil Analysis for Mining Equipment Maintenance	92
Chapter 11: Oil Analysis for Servicing On-the-Road Transportation Vehicles	94
Chapter 12: Oil Analysis for Offshore Drilling Reliability Service	96
Chapter 13: Oil Analysis for Municipal Water Treatment Maintenance	98
Chapter 14: Oil Analysis for Industrial Plant Reliability Management	100
Chapter 15: Oil Analysis for Reliability Service	105
Chapter 16: Oil Analysis for Engine Generator Maintenance	108
Chapter 17: Oil Analysis for Shipboard Maintenance	111

Section 1: Introduction

Chapter 1: In-service Oil Analysis for Condition Based Maintenance

The practice of in-service oil analysis or used oil analysis started over a century ago on locomotive engines. Now it is one of the most important components of condition based maintenance (CBM), a practice of assessing a machine's condition by periodically gathering data on key machine health indicators to determine maintenance schedules. Billions of dollars are spent every year replacing machinery components that have worn out due to insufficient lubricant performance. Knowing how to interpret changing lubricant properties can increase both the uptime and the life of mission critical assets. The existence or amount of debris and particles from wearing parts, erosion and contamination provide insights about the issues affecting performance and reliability.

Lubricants, fuels and other key fluids analyses provide critical early warning information indicative of machine failure. By analyzing and trending data, one can schedule maintenance before a critical failure occurs. The results are higher equipment availability and productivity, lower maintenance costs, lower total cost of ownership (TCO), fewer outages, optimal equipment performance and a greener operation.

What is In-service Oil Analysis?

Lubricating oil is the life blood of oil wetted machinery. In-service oil analysis can provide information about machine wear condition, lubricant contamination as well as lubricant condition (Figure 1). Reliability engineers and maintenance professionals can make maintenance decisions based on diagnoses of oil analysis results.

Machine wear analysis is essentially the analysis of particles in the oil. Machine wear can be classified into adhesive (sliding) wear, abrasive (cutting) wear, fatigue wear, and corrosive wear. A full suite of wear particle analyses includes measure of particle count and distribution, particle shape and morphology, wear metal and alloy elements

and the presence of large ferrous particles. Over the years different techniques and instruments have been deployed in the field or in commercial laboratories but essentially they all provide the information mentioned above. Even though all wear particle analysis techniques can be applied to different types of assets, specific issues are different for industrial rotating machines than from rotating engines. Reciprocal engines tend to generate fine wear particles and engine oils can be dark due to soot (nano-sized carbon particles as combustion byproduct). Elemental analysis is primary or sometimes the only analysis performed in a commercial oil lab that assesses engine wear conditions because concentrations of different wear metal elements indicate wear severity of moving parts in an engine. Slower moving rotating machines, such as gearboxes, tend to generate larger wear particles. As most of them are made of steel, large ferrous particle analysis is fairly common and easy to perform on oils from these assets.

Contaminants in oil can be in solid or liquid form. Solid contaminants such as sand and dirt are commonly monitored by particle counting and sizing techniques. Liquid contaminant for industrial rotating machines is mostly water. For diesel or gas engines, however, it can be water, coolant or fuel. A fairly common fluid contaminant across all asset types is the accidental or purposeful use of an incorrect lubricant when topping off old oil. All contaminants can significantly reduce the useful life of the oil and increase machine wear. They need to be prevented proactively with proper seals and filtration systems and they need to be monitored regularly.

Monitoring lubricant degradation helps one decide if the oil is no longer fit for use and if it needs to be changed. One key oil property is viscosity. The viscosity is typically measured at 40C for rotating machines and at 100C for engines. For oil in rotating machines, oxidation and acidity of the oil (Total Acid Number or TAN) are monitored to determine if the oil is still fit for use and to prevent corrosion. For engine oils, oxidation, nitration, sulfation and total alkaline additive reserve in oil (Total Base Number or TBN) are monitored. For natural gas engine oils, TAN is monitored along with other engine oil parameters.



Figure 1-1: Information provided by in-service oil analysis

Table 1 is a comparison of oil analysis parameters for engines and rotating machines.

Category	Parameters	Engines	Rotating Machines
Wear	Elemental concentration	Yes	Yes
	Particle sizes and shapes	Optional	Yes
	Large Ferrous particle concentration	Optional	Yes
Wear / Solid contaminants	Particle Count	No	Yes
Fluid Contaminants	Water	Yes, mostly dissolved water	Yes, dissolved and free water
	Glycol/Coolant contamination	Yes	Not applicable
	Fuel Dilution	Yes	Not applicable
	Fuel Soot	Yes	Not applicable
Degradation	Viscosity	40C and/or 100C	40C
	Oxidation	Yes	Yes
	Nitration	Yes	No
	Sulfation	Yes	No
	TAN	Yes for natural gas engine No for gas or Diesel engine	Yes
	TBN	Yes	No

Common oil analysis parameters for engines and rotating machines such as turbines, and gearboxes

Table 2 shows oil analysis parameters and the technologies used to measure them

Category	Engines	Rotating Machines
Machine wear	Fine wear metal elements	Rotating Disc Electrode (RDE) Spectroscopy*, Inductively Coupled Plasma (ICP) Spectroscopy
	Large wear metal elements	FPQ-XRF*, Acid digestion ICP
	Particle count and distribution	Light blocking*, light scattering, laser imaging (LNF)*
	Wear particle shape analysis	Laser imaging (LNF)*, Ferrography*
Contamination	Yes, mostly dissolved water	Yes, dissolved and free water
	Sand and Dirt	Light Blocking*, Light Scattering, Laser Imaging (LNF)*
	Fuel Dilution	Fuel Sniffer*, Gas Chromatography (GC), Gravimetric
	Water/Moisture	Infrared (IR)*, Karl Fischer Titration (KF)
	Glycol/Coolant	IR*
	Soot	IR*, gravimetric
	Alien Fluid	IR*
Degradation	Oxidation, Nitration, Sulfation	IR*
	Viscosity	Viscosity*
	Acid Number (AN), or Base Number (BN)	Titration, IR*

Key oil analysis parameters and corresponding analytical techniques. Techniques with an asterisk (*) are used in products from Spectro Scientific, Inc. with detailed explanations in the handbook.

Benefits of In-Service Oil Analysis

The key benefits of in-service oil analysis are maintenance cost savings and productivity increases from increasing the uptime of running machines. Machines run longer if the right oils are used and if the oils are dry, clean and fit for use.

Oil mix up is one of the most common lubrication problems contributing to machine failure. Putting the right lubricating oil in a machine is a simple task that can improve machine reliability. Checking the viscosity, brand and grade of incoming oil and checking for contamination of alien fluids help to reduce the chances of oil mix up and keep the machines running at optimal efficiency.

In order to keep the oil dry, clean and free of contaminants, seals and filtration systems need to work properly and oils should be checked regularly. Sand and dirt in oil cause the generation of abrasive wear. Moisture in oil causes corrosion. Fuel or coolant in engine oil changes viscosity and causes the generation of adhesive wear. It is necessary to keep the lubricating oil clean and dry at all times.

To keep oil fit for use, oil conditions are to be monitored regularly to make sure the oil is used within its performance specification. A well balanced oil analysis program shall monitor machine wear condition, oil contamination and oil degradation. Key parameters are measured regularly and their trends are closely monitored. If one or more parameters exceed the alarm limits or a change in the trending rate is detected, reliability engineers are alerted and maintenance actions may be needed to resolve potential problems.

The cost savings from a well-executed oil analysis program come from reducing production loss from unplanned down time due to catastrophic failure as well as decreasing repair costs and eliminating unnecessary oil changes. In a power plant or paper mill, the cost savings mainly come from reduced machine down time and repairs. In a mining site with hundreds of hauling trucks, the cost savings mainly come from eliminating potential engine failures. For a municipal transportation fleet, however, the cost savings from oil analysis are due to reduced material, labor and recycling by extending the oil drain intervals.

Onsite or Outsource – That is the Question

The awareness of oil analysis within the reliability professional community has increased over the years. Numerous case studies are published every year demonstrating how oil analysis and lubrication

management practices improve machine reliability and save money. Companies or organizations with large fleets of high value assets have established policies and processes using oil analysis in their predictive maintenance practices.

Outsourcing oil analysis to an offsite oil laboratory is common. Every year hundreds of millions of oil samples are analyzed by laboratories worldwide. Outsourcing oil analysis involves collecting oil samples from an asset and shipping them to a lab. Lab technicians then perform the requested oil analysis tests by asset type, and an analyst reviews the data and provides recommendations included on the final report. The report is then sent back to the user for review and, if needed, maintenance actions are performed based on the recommendations and maintenance schedule (Figure 2).

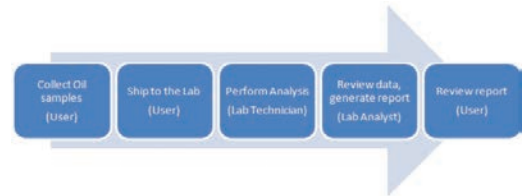


Figure 1-2: Off-site oil analysis flow chart

The advantage of outsourcing oil analysis is that an offsite commercial laboratory will have a complete set of oil analysis instruments and dedicated technicians to run the tests with experienced analysts to interpret the data. There is no upfront capital investment; therefore, the practice is simple to implement. It is ideal for a small- to medium-sized operation with a limited number of assets to test, or if oil analysis is a new practice for the organization.

The downside of outsourcing is the long turn-around time, from when the sample is collected to when the report is received. Typically, delay occurs during the collection or transportation of oil samples to the labs. Many labs have expedited services that can turn around a test report within hours after receiving oil. However, the oil bottles may have been sitting in a shipping hub for days, weeks, or even months before they are sent to the lab. So, a reliability engineer can rarely rely on lab reports to make decisions in time. While using other predictive maintenance tools such as infrared imaging camera, or vibration analyzer, equipment maintainers can make decisions and recommendations on-site in real time. Oil lab reports on the other hand are used to confirm a problem for post mortem analysis.

Even if an oil report points out a potential failure, the reliability engineers may have already missed the

window to service the machine, or a failure might have occurred before the report was received. This limits the effectiveness of oil analysis for any CBM program.

If an oil lab is set up on-site close to the assets and reports are provided within hours after oil sample is collected, then oil analysis reports can be part of the decision making process. On-site oil analysis is just another tool in the reliability engineer's toolbox, like vibration analyzers and infrared imaging cameras.

Things have progressed from years ago when setting up an on-site lab was an intensive project requiring renovations to lab space, additional staffing with high skill levels and months of time to implement. With the introduction of Spectro Scientific's turnkey oil analysis solutions, the capital expenditure, space, labor and skill requirements are significantly reduced. Spectro Scientific optimized each solution based on cost, throughput, ease of use and performance for different use cases. For example, the MiniLab Series was designed for industrial manufacturing and power generation plants testing 1000 to 3000 samples per year, while MicroLab was designed for testing 1000 to 3000 samples per year per shift for fleet operations such as mining, oil drilling, and on highway transportation. Both on-site labs do not require additional staffing or skilled operators. Space requirements are not more than a table top space. The costs of MiniLab and MicroLab are only a fraction of a full-sized oil lab. On-site oil analysis is part of the CBM process.

Onsite or outsource, that is the question. It ultimately depends on the answers to two questions:

- how critical is the consequence of the failure, and
- how many samples do you need to test annually?

For mission critical operations including the military, on-site analysis is a must. For plants or fleets with more than 500 samples per year, the benefit of doing on-site oil analysis quickly outweighs the cost using the MiniLab and MicroLab Series oil analyzers.

On-site oil analysis solutions

Oil analysis for different assets and applications can vary, not only because the test requirements are different between engines and rotating machines but because the working environments and the use of oil analysis results are different as well.

We will highlight five common applications here:

- Oil analysis for engine test labs as part of QA/QC process,
- On-site oil analysis for industrial manufacturing or power generation plants,

- On-site oil analysis for transportation, heavy mining or fleet managers in a service garage,
- Route-based (in the field) oil analysis for service engineers surveying assets in the field, and
- High volume, centralized industrial labs for large corporations or several mining sites in a remote location.

In all cases comprehensive oil analysis is required for engineers to better understand the condition of the assets, the condition of the oil and the condition of the filtration system. In summary, oil analysis systems are different for each application due to variations in requirements.

Engine Test Lab for R&D and Production Testing

Dyno testing is critical in the engine development, qualification and production process. Oil analysis for engine testing is an important tool in understanding engine performance. Comprehensive and near real time oil analysis can provide critical information about engine condition and the condition of the lubricant. During accelerated stress testing periods, oil analysis may be monitored at 10 to 15 minute intervals. This leaves limited time for engineers to perform the tests and analyze the results once the oil sample is collected though sample turnaround time is a high priority.

Some engine oil test labs include elemental analysis for trace wear metals, additive depletion and contaminants such as coolant leaks. They also include an infrared (IR) spectrometer to measure lubricant condition such as Oxidation, Nitration, Sulfation, Total Base Number (TBN), soot, moisture and coolant leaks as well as a fuel dilution meter to monitor fuel contamination in oil. Figure 3 is an example of engine test labs with the SpectrOil 100 Optical Emission Spectrometer (OES), FluidScan IR oil analyzer, and FDCheck fuel dilution meter.



Figure 1-3: Example of on-site oil analysis system for engine test labs including elemental OES, IR spectrometer and fuel dilution meter. The system does not require sample preparation, or chemicals for cleaning. In addition, only 3 ml of oil is consumed for all three tests.

Portable Q5800 for Field Service Professionals

It is not always easy to collect oil samples in the field and bring them to a lab for analysis. Engineers on an offshore drilling rig sometimes have to wait weeks or months to have the samples delivered to an onshore lab. In addition, there may be space restrictions or resources limitations to performing oil analysis on-site, especially if reliability services are outsourced to a third party service company. In this case, a team of service engineers go on-site periodically for testing, that includes oil analysis using portable tools. Technology advancements now make it possible to have portable, battery powered oil analysis tools with analytical capabilities comparable to the instruments used in laboratories. Now reliability service engineers can combine the information from oil analysis and vibration analysis in real time. This empowers them to make much informed decisions and recommendations about machine maintenance. Figure 6 shows a FieldLab 58, which includes a particle counter, a X-ray Fluorescence (XRF) detector for 13-element wear particle analysis, a direct IR spectrometer for oil chemistry and a temperature controlled kinematic viscometer. All the technologies are packaged in a suitcase and powered by a lithium ion battery for up to four hours of continuous testing. Weighing only 35 pounds, reliability service professionals can carry it to their critical asset for just in time oil analysis. Data from the FieldLab 58 Series can be uploaded wirelessly to the SpectroTrack LIMS software so an analyst or manager thousands of miles away can review the data and reports in real time.



FieldLab 58 Expeditionary Fluid Analysis System

Full size Industrial Tribology Lab (ITL) for Commercial or Industrial Laboratories

When laboratory grade analytical instruments are needed to manage large sample volumes, a turnkey Industrial Tribology Lab (ITL) is recommended. An ITL is a complete set of oil analysis instruments suitable for performing the most demanding in-service oil analysis tasks commonly performed in a commercial laboratory (Figure 7). Instruments in ITL are designed for small foot print, no or little sample preparation, low consumable cost, low waste stream, and easy to operate. It is commonly used for remote mining sites, railway repair depot, shipboard, or the central lab in an industrial plant.

The core instrumentation in an ITL includes a Rotating Disc Electrode (RDE) Optical Emission Spectrometer (OES), a laser imaging particle analyzer based on LaserNet Fines® technology, a high speed automatic viscometer, an infrared spectrometer and SpectroTrack LIMS software.

ITL configuration with a sample SpectroTrack LIMS report



Table 3 is a detailed summary of five different on site oil analysis solutions including intended applications, annual sample volume and detailed analytical test parameters.

	FieldLab 58	MicroLab 40	MiniLab 153	Engine Test Lab	Industrial Tribology Lab
Applications	Field Service tool	Fleet management Screening tool	Industrial & power plants	Engine testing (Dyno)	Mining, power plants, industrial plant central lab
Typical # of Samples/Yr	1000 - 3000	1000 - 3000	1000 - 5000	1000 - 20000	5000 - 20000
LIMS software	Subscription to SpectroTrack.com	Subscription to myLubeTrak.com	OilView LIMS	Not available	Local or hosted SpectroTrack
Elemental Analysis	Yes. 13 elements for wear particles > 5um. Manual	Yes. Up to 20 elements for particles < 10um, automatic	Yes. 24 elements for particles less than 10um, manual	Yes. 24 elements for particles less than 10um, manual	Yes. 24 elements for particles less than 10um, manual. Automatic version optional
Viscosity	Visc @ 40C, manual	Visc @ 40C & 100C, automatic	Visc @ 40C, manual	Optional	Visc @ 40C & 100C, automatic
Oxidation	IR, Manual	IR, Automatic	IR, Manual	IR, Manual	IR, Manual
Nitration	IR, Manual	IR, Automatic	IR, Manual	IR, Manual	IR, Manual
Sulfation	IR, Manual	Not Available	IR, Manual	IR, Manual	IR, Manual
TAN	IR, Manual	Not Available	IR, Manual	IR, Manual	IR, Manual
TBN	IR, Manual	IR, Automatic	IR, Manual	IR, Manual	IR, Manual
Water	IR, dissolved water only, Manual	IR, dissolved water only, automatic	IR, total water, Manual	IR, dissolved water only, Manual	IR, total water, Manual
Soot	IR, Manual	IR, Automatic	IR, Manual	IR, Manual	IR, Manual
Antiwear additive Depletion	IR, Manual	Not Available	IR, Manual	IR, Manual	IR, Manual
Glycol contamination	IR, Manual	IR, Automatic	IR, Manual	IR, Manual	IR, Manual
Fuel Dilution	N.A.	IR, Gasoline only, automatic	N.A.	Fuel Sniffer, Manual	Fuel Sniffer, Manual
Particle Count	Yes, # of p/ml > 4um, pore blockage	ISO 4406, light blockage	ISO 4406, Laser Imaging	N.A.	ISO 4406, Laser Imaging, Auto Sampler Optional
Particle shape classification	N.A.	N.A.	Yes	N.A.	Yes, Auto sampler optional
Ferrous particle analysis	N.A.	N.A.	Yes, large Ferrous & Total Ferrous	N.A.	Yes, large Ferrous & Total Ferrous, Auto sampler optional

Summary of on-site oil analysis systems for various applications

Conclusion

In summary, in-service oil analysis provides critical information about machine condition and oil condition. There are various approaches to implementing an oil analysis program, depending on the application and maintenance objectives. Modern technologies have given reliability professionals new tools they never had before for effective maintenance based on information from in-service oil analysis.

Chapter 2: Oil Sampling Best Practices

Introduction

Obtaining a representative oil sample from a closed loop lubrication system is essential to any good oil analysis program. As oil analysis programs become more standardized for routine monitoring, especially within equipment warranty support programs, equipment makers provide more sampling points on new equipment. These access sampling points make it easier for operators to quickly and easily take representative oil samples. This article outlines an overview of the theory behind obtaining consistent oil samples, current accessories for oil sampling, and offers guidance on when and where to sample (if your equipment does not have a OEM supplied point).



Figure 1: Sampling points preinstalled on a CAT 320D excavator. Caterpillar SOS program relies on good oil analysis sampling to ensure warranty programs are effective. (ref: Spofford Construction)

Understanding Dynamic Particle Equilibrium in Closed Loop Systems

Dynamic Equilibrium Condition (DEC) is defined as a steady state condition where the normal wear rate in a machine results in no net gain or loss of particles. Knowing what that level is in any lubrication system is necessary in order to detect departures from this level as a result of an abnormality. At equilibrium operating conditions, the rate of wear particle generation is constant. Under the same conditions, the particle separation and removal rates, although size dependent, are also constant, surprisingly, with or without a filter. Not surprisingly, the population of all but the smallest particles reaches an equilibrium level. Departure from that normal level is an indication of an abnormality in the system; perhaps the filter is bypassing or has failed, or a severe wear mode has begun. The smallest particles remain in the system, suspended and unaffected by the filter, so their population tends to increase during the life

of the oil. Therefore, very fine particles are composed of the oldest particles, some of which may have been generated as larger particles, but have been reduced in size by various chemical and physical processes. The larger particles are relatively new and are more representative of the current condition of the wearing surfaces. The particles in an oil sample tend to settle out. If they are large and dense, they settle out rapidly. The particles of wear, corrosion, oil degradation, and contamination provide valuable diagnostic information about the condition of the oil as well as the condition of the wearing surfaces of the machine. Since particles exist as a separate phase in the oil, they are not evenly distributed in the system. All the freshly made wear particles will be present immediately after a wearing mechanism such as a roller bearing, gear, sliding surface, etc. The largest metal particles, which are of such critical interest to the analyst, are soon removed by settling in the sections with slow moving oil, such as the sump, or they are filtered or otherwise separated. The very smallest particles tend to remain suspended and pass through all but the finest filters so they are generally distributed evenly throughout the oil piping system. Consequently, in order to capture a representative sample, the sampling location must be carefully considered (Figure 2).

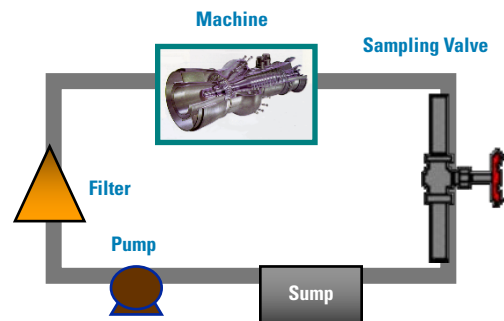


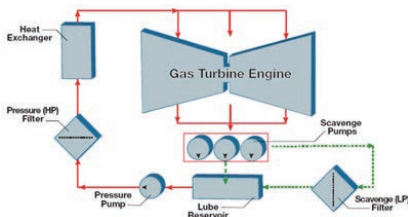
Figure 2: Typical closed loop lubrication system

Sampling in a Closed Loop System

The best primary location to monitor in a closed loop system is AFTER the area of greatest wear/stress, and BEFORE the filter. This may be before the return line filter housing, or in the reservoir at the return line zone. Secondary sampling points may be chosen also, such as AFTER the filter, and may be used to evaluate filtration efficiency (Figure 3). Another sample point is the sump or reservoir.

When sampling from lubrication lines, the sample should be taken from the “isokinetic line of flow” in the line. In other words the velocity of the oil at the point of sampling should be equal to the velocity of the oil at the midpoint of the line. A common sampling point to achieve this is on the elbow of the return line (Figure 4). Where and how to sample hydraulic lines is detailed in the ISO 4021 for hydraulic systems.

A recent advance in sampling technology is to install “sample boxes” on equipment with multiple lubricant compartments and/or other fluids to be sampled, such as coolant, or DEF fluid. Sampling lines are installed and a junction box created so that an operator can sample all compartments with a dedicated point a mileage or hourmeter is installed for record keeping. This approach is gaining traction with larger mining equipment such as excavators and large trucks (Figures 5 and 6).



Primary sampling point (A) Secondary Point(s) (B)

Figure 3: A Gas turbine lubrication schematic. Primary sampling points is the return line before reservoir, and secondary is before and after the HP filter. Ref: Pall Corp

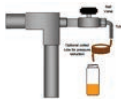


Figure 4: Sampling from an elbow on a hydraulic line per ISO4021. Pall Corp



Figure 5 and 6: Fluid Sampling station for mining equipment (Ref: Fluid transfer Systems Welshpool Australia)

Sampling from Pipes

- The pipe ideally will contain oil soon after draining from the wearing part.
- The flow rate should ideally be turbulent to keep particles entrained in the oil.
- The oil should be hot; do not sample after cooling.
- If the pipe is large and the flow is laminar, take a sample from near the center of the pipe to avoid the lower wall where old debris may accumulate.
- If the sample valve is installed with a dead leg line, be sure to flush thoroughly before collecting a sample.

Sampling from Tanks

- Take the sample from near the center and well clear of the bottom or the sides. Use a pitot tube attached to a sampling port installed on the sidewall to ensure the sampling area of interest is addressed. Be mindful of the extra dead leg in the pitot tube line – this must be flushed before the bottle is filled.
- Sometimes a special spring steel sample stick can be useful in placing the suction tube just where you want it each time, thereby gaining repeatability. The narrow flat steel bar can be fitted with a standoff to prevent bottom sampling, as well as clasps to hold the Tygon tubing.

Sampling Techniques for Oil Analysis

Sampling Test Ports

Sampling test ports are now the default standard for obtaining liquid oil samples from both reciprocating engines, hydraulic systems and rotating equipment (turbines, compressors, gearboxes, motors). Almost all new equipment has preinstalled sampling valves (Figure 7), particularly when vendors insist on oil analysis as part of the warranty program.



Figure 7: Options available for sampling pressurized valves (Ref: Checkfluid)

Pressurized Probe

The most common and popular style for general industrial and hydraulic sampling needs is the female test port designed to adapt a sampling probe. The brass or stainless steel valves (Figure 8) have either elastomer seals or metal ball seat designs, able to withstand up to 5 MPa (750 psi sampling pressure dependent on the style). They normally come with JIC fittings, (SAE J514) that are standard for these applications. These ports are preferred for industrial applications because the valve accepts a large (4 mm) probe, ideal for sites where oil samples with high viscosities are encountered, and they also can be used with sampling thief pumps when installed on non-pressurized bath lubricated reservoirs, such as gearboxes.



Figure 8: Using a sampling pump with a probe adapter (Ref: Checkfluid)



Figure 9: Sampling a Jenbacher gas engine with a needle probe valve (Ref : GE)

Probalizer/ SOS style Needle Probe systems

A similar type of pressurized sample port primarily used for reciprocating engines and transmissions in fleet applications is the needle valve (2 mm) test port (Figure 9). A small disposable needle valve is connected to the sampling bottle, providing a simple one step tool for drawing an oil sample quickly from an engine (Figure 10).



Figure 10: Probalizer needle probe and bottle cap (Schroeder Industries)



Figure 11: Push button sample valves (Ref: checkfluid)

Other variants in the market include push button valves. (Figure 11) These drain valves are used for oil analysis of reservoirs or sumps. Recently a new technology has become available called Luneta condition monitoring pods, These combine a sampling port/tap and oil sight glass into one (Figure 12). These devices may be retrofitted to reservoirs and sumps to replace older sight glasses, and avoid the need for a separate sampling port.



Figure 12: Condition monitoring pod (Ref: luneta)



Figure 13: Sampling thief pump, (ref: Easy Vac)

Non Pressurized manual sampling

Older equipment (pre-1980 build) rarely had sampling valves installed, and so it was up to the maintenance personnel to configure a sampling point and methodology. Sampling thief pumps (Figure 13) with Tygon tubing have been used for many years to sample reservoirs manually. The pumps are designed to create a vacuum in the bottle and cause the oil to leave a non-pressurized reservoir (Figure 14).

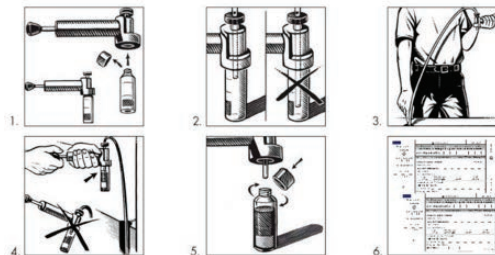


Figure 14: Sampling from reservoirs using a thief pump. the sampling bottles are designed to fit to the pumps, a 38mm round bottle mouth is very common. (Ref :Shell Lube analyst User Guide)

Bottles

Sampling bottles come in many shapes and sizes. 120 ml (4 oz) HDPE (opaque) bottles have been the most common sampling bottle for many years. Many suppliers now provide clear PET, or PS bottles for oil sampling so end users can readily see debris or water in the sample, forcing a resample before sending it over to the testing area, or outside service provider (Figure 15).

Super Clean bottles

Bottles that are cleaned with filtered air are sold with a designation “Super clean” and are designed for applications where particulate contamination is of utmost concern. Sometimes these bottles come with a seal wrapper between the mouth and the cap. There is no standard for what is considered “clean” so it is up to the buyer to clarify what the cleanliness level is. These usually cost more than regular sampling bottles, and are somewhat ineffective, as care is needed since the bottle is contaminated immediately upon opening the cap. Some operators minimize the ingress of debris in plant environments by sampling within a polyethylene bag, however this can get very messy as the bag makes it easy for the operator to drop the sample.

UCVD

Ultra Clean Vacuum Device bottles are cleaned to an ISO code of 11/9/4 and sealed. Unlike other “super clean bottles” there is no need to open the cap. The bottle may be used in conjunction with a sampling probe or needle valve, and it also avoids the need for a sampling thief pump. The operator simply connects the tubing from the port to the bottle and opens the valve. When finished, bottle valve is closed and the cap is replaced on the UCVD (See Figure 16).



Figure 15: Opaque (HDPE) and clear (PETE) 120 ml sampling bottles (ref: Apex oil lab)



Figure 16: Sampling process using UCVD bottles (Ref: Mecoil)

Labeling sampling points and samples

Misidentified sample points and samples are a very common source of confusion. All sampling points, once identified, should be properly identified with a sign, detailing the sample point code as defined by the organization's asset management system, and the oil brand and grade, at a minimum (Figure 17).



Figure 17: A properly labeled sampling point (Ref: Trico)

Use preprinted asset labels from your asset management software, LIMS or service provider.

- Immediately label samples. Misidentified samples are a very common source of confusion.
- Use preprinted asset labels from software (e.g. OilView, SpectroTrack) where possible (Figure 18). The more information present, the more meaningful the sample results will be.



Figure 18: Oil sample bottle labels preprinted with OilView software (Ref: Emerson Process Management)

Dos and Don'ts of Sampling

- DO Sample when machine is operating, or within 30 minutes of doing so.
- DO Sample from same point always.
- DO immediately label the sample to prevent mix-ups
- DO drain off dead leg oil/water/debris before sampling.
- DO NOT sample immediately after oil change or makeup.

Sampling Frequency

Machinery manufacturers will often suggest a sampling interval, but that should only be a rough guideline. The equipment asset owner is the best judge of sampling intervals. Pertinent questions in arriving at a sampling interval include:

- Safety Risk (i.e. loss of life or limb if catastrophic failure occurs)
- Criticality of equipment (or lack of redundancy)
- Environment (wet, dry etc.)
- Operating conditions (load, speed)
- What is the failure history?
- How costly is a failure? In repair cost? Lost production? Life and safety?
- Have operating conditions changed to put more stress on the machine?

In general, a quarterly or monthly sampling interval is appropriate for most important industrial machinery, whereas reciprocating engines tend to be sampled at a more frequent interval based on run time on the oil and engine. The answers to the above questions will help decide which regimen is more appropriate. In a new program, it usually makes sense to start with a monthly interval and then extend it as experience dictates. Having onsite oil analysis equipment allows the user to self-manage and extend intervals safely as trends develop.

Further guidance may be sought by consulting with the equipment manufacturer and oil supplier. The following is designed as a guide to establish proper intervals.

Industrial and Marine			
Equipment Type	*Normal Use Sampling Frequency (Hours)	Monthly (Calendar)	Occasional Use Sampling Frequency (Calendar)
Steam Turbines	500	Monthly	Quarterly
Hydro turbines	500	Monthly	Quarterly
Gas Turbines	500	Monthly	Quarterly
Diesel Engines-Stationary	500	Monthly	Quarterly
Natural Gas Engines	500	Monthly	Quarterly
Air/Gas Compressors	500	Monthly	Quarterly
Refrigeration Compressors	500	Monthly	Quarterly
Gearboxes-Heavy Duty	500	Monthly	Quarterly
Gearboxes-Medium Duty		Quarterly	Semi-Annually
Gearboxes-Low Duty		Semi-Annually	Annually
Motors-2500 hp and higher	500	Monthly	Quarterly
Motors-200 to 2500 hp		Quarterly	Semi-Annually
Hydraulics		Quarterly	Semi-Annually
Diesel Engines-On and Off Highway	150 hours/10,000 miles	Monthly	Quarterly

Off-Highway/Mobile Equipment	
Equipment Type	*Normal Use Sampling Frequency (Hours/Miles)
Gasoline Engines	5000 miles
Differentials	300 hours/20,000 miles
Final drives	300 hours/20,000 miles
Transmissions	300 hours/20,000 miles

Aviation	
Equipment Type	*Normal Use Sampling Frequency in hours
Reciprocating Engines	50 hours
Gas Turbines	100 hours
Gearboxes	100 hours
Hydraulics	100 hours

Training

Even with improved sampling ports and easier access, it is still the responsibility of the operator to take a proper sample. Best in class organizations should have a standard work instruction for each class of equipment, and if the sampling point is in a confined space, or close to rotating components, additional safety procedures such as the lockout/tag-out system enforced. A person experienced in oil sampling should walk through a process with the trainee, advising him on each machine to be sampled. Follow up should be done with documentation on the method of sampling and the necessary tools.

Sampling Routes and Use of At-line Oil Analysis Tools

Inspection route-based vibration analysis and thermography are standard methods of machinery health monitoring, and are now possible with oil condition monitoring. Machine oil condition monitoring yields useful and critical information on machinery health, and complements the data provided by vibration analysis.

Until now, machinery inspection routes have not been able to meaningfully integrate oil condition monitoring. Only handheld data recorders were used to log lubrication management metrics such as the quantity of oil added, or the lubricant level. Actual monitoring of the oil condition

was not performed because there was no technology available that provided accurate, meaningful information quickly and easily. With the FluidScan™ Q1100, it is feasible to obtain critical, quantitative oil parameters right at the sampling point, in one minute, from a single drop of oil. As a result, it is now possible to implement a route-based machinery oil condition analysis work process (Figure 19).

The FluidScan can download all of the necessary information (Area/Equipment/Point, Oil type, and Alarm Limit sets) from the Emerson OilView program to create a route. This saves time and ensures the correct data is entered for each piece of machinery. Once the route is completed, the FluidScan measurement data for each point sampled on the route is imported into AMS so a comprehensive asset database is maintained.

Route-based oil analysis provides greater flexibility for on-site oil analysis programs, and provides the potential to streamline work flow processes within an industrial setting with an easy to use instrument.

Figure 19: Operators sampling and testing oil condition of a hydraulic reservoir (Ref: Spectro scientific)



Conclusion

Oil analysis programs are only effective if the sample is representative of the equipment condition, and the technique and sample location are not variable. Good input supports good output. New and existing program owners have a wide range of support and technologies available today to ensure sampling is consistent, easy and reliable.

References

Checkfluid.com, Fluid transfer systems.au, Schroeder Industries, Noria.com, Luneta.com, Pall Corp, Apex Oil Lab, Trico, Jenbacher, Emerson, Mecoil, Shell LubeAnalyst

Barracough T “Establishing Wear Particle Limits Using the Theory of Dynamic Equilibrium Condition (DEC) to Predict when Abnormal Wear Modes are Taking Place in Machinery”

Section 2: Technologies

Chapter 3: Elemental Analysis

Elemental analysis remains the pre-eminent technology for oil analysis. Its multi-faceted capability to probe contamination, additives, and pinpoint machinery wear in a quantitative fashion is unmatched by any other type of analysis. The two key use modes of the technology are for lubricant blenders, who must carefully monitor the amount of additives in the blend, and for the determination of machinery health. Elemental analysis has kept ahead of the growing improvements in machinery, including filtration, which continue to drive down the overall level of debris material in the oil. This capability allows the technology to provide appropriate solutions for the entire range of machinery, from jet aircraft to Class 8 trucks. Its key operating principal for machinery analysis is that specific elements (20+) of interest in the oil are quantified into ppm levels as the machinery is operated. "Of interest" means the range of elements that physically comprise that machinery component and so may be lost to the oil as debris, as well as additives in those components' oil such as Zinc (Zn), Calcium (Ca) and Phosphorus (P), and elements that mark contamination such as Silicon (Si) from dirt. Based on guidance for the particular machinery component being monitored, the real-time amount and the trend levels for each element are examined and maintenance actions are taken as indicated. For example, Iron (Fe) or Copper (Cu) levels exceeding 200 ppm in a sample of city bus engine oil may indicate that bearings are wearing at an unacceptable rate and the engine should be removed from service, or a cruise ship operator may change a 500 gallon oil sump when the additive levels of Ca and P are down to ½ their original level.

The two core elemental analysis techniques for machinery analysis continue to be Rotating Disc Electrode (RDE) and Inductively Coupled Plasma (ICP)-based Optical Emission Spectrometry (OES) systems. RDE and ICP describe ways in which to vaporize the material in the oil. Upon vaporization of the material, the elements present emit light at their characteristic wavelengths, which are detected by the OES system and quantified into ppm levels. The SpectrOil product line from Spectro Scientific has for decades been the premier solution for RDE, which is the solution of choice when rapid and no sample preparation analysis is needed.

These core techniques have always relied on the assumption that the machinery being monitored will

generate a continuum of debris, as neither will be able to probe particulate that is greater than approximately 10 microns in circular diameter (5 microns for ICP, 10 microns for RDE). This is due to the fact that the vaporization energy needed to do so is too high for such large particles. When the small particle assumption does not hold, special techniques such as acid digestion must be employed in order to further break down the debris to measurable sizes before sample analysis. As overall debris levels in the oil continue to decline in general, analyzing such particles becomes increasingly more important: Spectro Scientific has developed the Q5800, which provides for a new means of elemental analysis which focus on the so-called "gap" in sensitivity between 10 microns for RDE and 50 microns for microscopy-based specialty solutions (such as Scanning Electron Microscopy/Energy Dispersive X-Ray analysis (SEM/EDX)). The Q5800 employs a two-step process of pre-concentration of particulate >4 microns in the neat oil on-to a filter coupon, and subsequent analysis of the particle by X-Ray Fluorescence (XRF). This technique is extremely sensitive (sub-ppm) to debris across the gap region and beyond. Further, increases in the generation rate of such large particulate in and of itself, captured by the Q5800, is a direct measure of the approach to machinery breakdown. In many monitoring situations, it is only the presence of such large particulate that will trigger an alarm.

Finally, alternatives to RDE/ICP analysis may also be utilized when the use case indicates the need. A prominent example is in the MicroLab® Series from Spectro Scientific. The core principle of the device is that the single touch of a button initiates an entirely autonomous analysis sequence. In this case, OES is effectively performed via utilization of a fountain electrode, rather than with a rotating disc (RDE). As for RDE, in this fountain electrode system a spark is generated between 2 electrodes with a narrow gap between them, which vaporizes the debris-containing oil. However, the oil in a fountain electrode emerges via a pump through the bottom electrode itself, which is meant to be permanent and have the ability to be operated repeatedly. In this way, autonomous elemental analysis may be performed.

3.1: Rotating Disc Electrode OES (RDE-OES)

Introduction

The basis of modern oil analysis programs is the use of optical emission spectroscopy (OES) to measure the ppm (parts per million) levels of wear metals, contaminants, and additives in oil samples. Whatever an oil lab may need to measure, multi-elemental analysis is the core of an in-service oil analysis program. This paper gives an overview of Rotating Disc Electrode Optical Emission Spectroscopy (RDE-OES), its applications, and the SpectrOil Series family of products, which combine the latest innovations for increased performance and reliability with 30+ years of experience in military and laboratory applications.

Early Background

Sometime after World War II, the Denver and Rio Grande Railroad, now defunct, began analyzing diesel locomotive engine oil by looking at the spectral lines emitted by an in service oil sample when excited by a strong electric arc using carbon electrodes. Early tests proved that monitoring the elements attributed to wear and contamination provided early warnings of chronic equipment failure.

Elemental spectroscopy is the backbone of an oil analysis program as it is widely applicable to a closed loop lubricating system such as those found in gas turbines, diesel and gasoline engines, transmissions, gearboxes, compressors and hydraulic systems. In practice, an oil sample is periodically taken from a system and analyzed. The resulting data, when compared to previous analyses and allowable limits, may indicate a sound mechanical system showing only normal wear, or it may point out a potentially serious problem in its early stages. With this advanced warning, steps may be taken to correct the situation before serious damage or injury occurs.

Spectroscopic oil analysis works by detecting the fine particles generated by relative motion of metallic parts in an oil-wetted system. Contaminants are also detected and lubricant mix-ups, or badly degraded lubricants, are identified by the concentration of additive elements. Multielement analysis, coupled with knowledge of the materials used to build the engine, often allows identification of a specific component in distress. Table 1 shows that typical metal elements can be analyzed by spectroscopy and their typical sources.

Metal	Engine, Transmission, Gears	Hydraulic Fluid	Coolants
Aluminum Al	Pistons or Crankcases on Reciprocating Engines, Housings, Bearing Surfaces, Pumps, Thrust Washers	Pumps, Thrust Washers, Radiator Tanks	Coolant Elbows, Piping, Thermostat, Spacer Plates
Barium Ba	Synthetic Oil Additive Synthetic Fluid	Additive	Not Applicable
Boron B	Coolant leak, Additive	Coolant Leak, Additive	pH Buffer, Anticorrosion Inhibitor
Calcium Ca	Detergent Dispersant Additive, Water Contaminant, Airborne Contamination	Detergent Dispersant additive, Water Contaminant, Airborne Contamination	Hard Water Scaling Problem
Chromium Cr	Pistons, Cylinder Liners, Exhaust Valves, Coolant Leak from Cr Corrosion Inhibitor	Shaft, Stainless Steel Alloys	Corrosion Inhibitor
Copper Cu	Either brass or bronze alloy detected in conjunction with Zinc for brass alloys and Tin for bronze alloys. Bearings, Bushings, Thrust Plates, Oil Coolers, Oil Additive	Bushings, Thrust Plates, Oil Coolers	Radiator, Oil Cooler, Heater Core
Iron Fe	Most common of wear metals. Cylinder Liners, Valve Guides, Rocker Arms, Bearings, Crankshaft, Camshaft, Wrist Pins, Housing	Cylinders, Gears, Rods	Liners, Water Pump, Cylinder Block, Cylinder Head
Lead Pb	Bearing Metal, Bushings, Seals, Solder, Grease, Leaded Gasoline	Bushings	Solder, Oil Cooler, Heater Core

Magnesium Mg	Housings on Aircraft and Marine Systems, Oil Additive	Additive, Housings	Cast Alloys
Molybdenum Mo	Piston Rings, Additive, Coolant Contamination	Additive, Coolant Contamination	Anti-cavitation Inhibitor
Nickel Ni	Alloy from Bearing Metal, Valve Trains, Turbine Blades	Not Applicable	Not Applicable
Phosphorous P	Anti-wear Additive	Anti-wear Additive	pH Buffer
Potassium K	Coolant Leak, Airborne Contaminant	Coolant Leak, Airborne Contaminant	pH Buffer
Silicon Si	Airborne Dusts, Seals, Coolant Leak, Additive	Airborne Dusts, Seals, Coolant Leak, Additive	Anti-foaming and Anticorrosion Inhibitor
Silver Ag	Bearing Cages (silver plating), Wrist Pin Bushings on EMD Diesel Engines, Piping with Silver Solder Joints from Oil Coolers	Silver Solder Joints from Lube Coolers	Not Applicable
Sodium Na	Coolant Leak, Salt Water and Grease in Marine Equipment, Additive	Coolant Leak, Salt Water and Grease in Marine Equipment, Additive	Inhibitor
Tin Sn	Bearing Metal, Piston Rings, Seals, Solder	Bearing Metal	Not Applicable
Titanium Ti	Gas Turbine Bearing Hub Wear, Turbine Blades, Compressor Discs	Not Applicable	Not Applicable
Zinc Zn	Anti-wear Additive	Anti-wear Additive	Wear Metal from Brass Components

Table 1: Typical source of elements analyzed by spectroscopy

Principles of Spectroscopy

Spectroscopy is a technique for detecting and quantifying the presence of elements in a material. Spectroscopy utilizes the fact that each element has a unique atomic structure, and when subjected to the addition of energy, each element emits light of specific wavelengths or colors. If this light is dispersed by using a dispersing element, such as a prism, a line spectrum will result. Since no two elements have the same pattern of spectral lines, the collected light can be analyzed and each element contained in the sample identified. Additionally, the intensity of the emitted light is proportional to the quantity of the element present in the sample, allowing the concentration of that element to be determined.

These spectral lines are unique to the atomic structure of only one element. For the hydrogen atom, with an atomic number of 1, the spectrum is fairly simple (Figure 1). On the other hand, the spectrum of iron with an atomic number

of 26 is much more complicated with many emission lines in the visible spectrum corresponding to the many possible electronic transitions that may occur (Figure 2). If more than one element is present in the sample, spectral lines of distinctively different wavelengths will appear for each element. These lines must be separated in order to identify and quantify the elements present in the sample. Usually only one spectral line among many is chosen to determine the concentration of a certain element. This line is chosen for its intensity and freedom from spectral line interference of other elements. To accomplish this, an optical system is required.



Figure 1: Emission Spectrum of Hydrogen



Figure 2: Emission Spectrum of Iron

Rotating Disc Electrode Optical Emission Spectroscopy (RDE-OES)

Spectrometers that look at the multitude of spectral lines from a heated, or “excited,” sample are called optical emission spectrometers. All optical emission spectrometers consist of three main components.

- 1. Excitation Source** – introduces energy to the sample.
- 2. Optical System** – separates and resolves the resulting emission from that excitation into its component wavelengths.
- 3. Readout System** – detects and measures the light that has been separated into its component wavelengths by the optical system and presents this information to the operator in a usable fashion.

One typical method used in the excitation source in modern spectrometers is an electric discharge. The source is designed to impart the energy generated in an arc, or spark, to the sample. For oil analysis spectrometers, a large electric potential is set up between a disc and rod electrode with the oil sample in the gap between them. An electric charge stored by a capacitor is discharged across this gap creating a high temperature electric arc, which vaporizes a portion of the sample, forming a plasma. A plasma is a hot, highly ionized gas which emits intense light. The light given off as a result of this process contains emissions from all the elements present in the sample. These emissions can now be separated into individual wavelengths and measured using a properly designed optical system. Temperatures in the 5000 to 6000°C range are achieved and even difficult to excite elements emit enough light to be readily detected.

Since the early days of spectroscopic oil analysis, oil has been sparked, or “burned,” between a rotating carbon disc electrode and a carbon rod electrode. The sample is placed in a sample cap, the disc is partially immersed in the oil sample and the disc rotates as the burn proceeds (Figure 3). This requires about 2 or 3 ml of sample, depending on the exact cap being used.

Figure 3: RDE spectrometer sample stand showing oil sample being “burned”



A fresh disc and a newly sharpened rod are required for each sample to eliminate sample carryover. This method is called rotating disc electrode (RDE) optical emission spectroscopy (OES), or combining the two, RDE-OES. Alternatively, it is referred to as RDE-AES, which stands for rotating disc electrode atomic emission spectroscopy. The light coming from the plasma is separated by the optical system, in a spectrometer, into the discrete wavelengths that comprise it. An optical device called a diffraction grating, is used to separate the discrete wavelengths. The diffraction grating is a concave mirror with very fine lines on its surface that causes incident polychromatic light to be separated into component wavelengths and focused on an array of light detectors.

Figure 4 shows the major components of an oil analysis spectrometer using a polychromator optic, based on the Rowland Circle concept. Light from the excitation process, or the “burn,” exits the fiber optic cable and passes through the entrance slit, where it is concentrated on the diffraction grating by a lens. The entrance slit introduces light made up of all the elements present in the oil sample and defines the shape of the spectral lines at the focal curve after it is diffracted by the grating. The purpose of the grating is to separate, or “diffract,” this light into its component wavelengths. The spectral lines can be photographed or electronically quantified by photomultiplier tubes (PMTs) or charge coupled devices (CCDs).

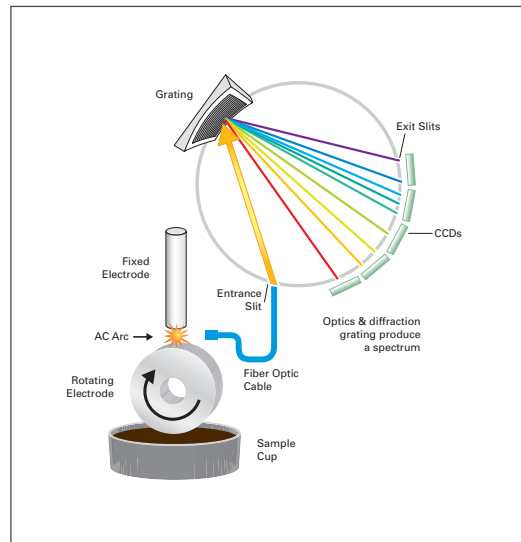


Figure 4: Schematic of a Rotating Disc Electrode Optical Emission Spectrometer for Oil Analysis

The readout system of a spectrometer is typically controlled by an industrial grade processor and software. A clocking circuit and amplifier periodically read the charge on a Photo Multiplier Tube, or CCD chip, and convert it from an analog to a digital (ADC) signal to measure the light that has fallen on a pixel. The charge accumulated on a pixel is converted to an arbitrary number defined as “intensity” units. At the end of the analysis, the total intensities for each element are compared to calibration curves stored in memory, and are converted to the concentration of the element present in the sample (Figure 5). Concentration is usually expressed in parts per million (ppm). This information can either be displayed on a video screen, or printed. Once the analysis is completed and the results are recorded, the system is ready for the next analysis. The analysis results may be left on the screen, stored on the hard disk, or sent to an external computer.

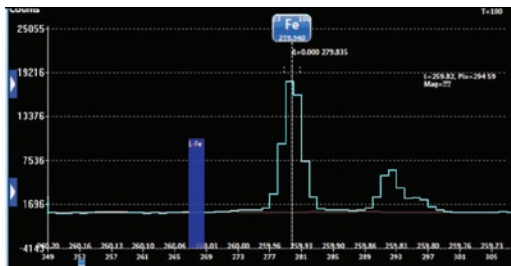


Figure 5: Readout system of an RDE-OES

SpectrOil Family of Spectrometers: The Market Leading RDE Spectrometers

RDE-OES spectrometers for used oil analysis continue to be the choice for elemental measurement. It has been more than 50 years since the technology was first used by railways and the military because of the instrument’s ruggedness, reliability, and stability. Research and development continues with the technique, with major improvements in limits of detection, and long term stability, a result of new electronic hardware, software and optics. The latest innovations in these areas are incorporated into the SpectrOil 100 Series spectrometer, shown in Figure 6. This system has a very small footprint, and is easier to use than ever before, making it the solution of choice for lubricant, coolant, and on-site fuel labs within mining, power generation, and commercial laboratories. The SpectrOil is available in a variety of application specific configurations shown in Table 2.

The SpectrOil M is a rugged and transportable system primarily used for military applications as specified by the DoD JOAP program (Figure 7). SpectrOil 110E is tailored to the specific requirements of engine monitoring, while the SpectrOil 120C fulfills the requirements of ASTM D6595, and is the default option for commercial customers.¹ The SpectrOil 120F for fuel analysis meets the requirements for ASTM D6728, and an additional L/D program is available to meet the specific low vanadium and alkali element limits required by GE Power systems gas turbines, per the GE- MTD-TD-002 specification. These systems are also used for coolant analysis and washdown water analysis.²



Figure 6: The SpectrOil 100 Series RDE Spectrometer



Figure 7: SpectrOil M Series elemental analyzer

APPLICATION	110E BASIC ENGINE		120C		120F FUELS
		STANDARD LUBRICANTS	EXTENDED OPTION	COOLANT OPTION	
Ag	0 - 900	0 - 900			
Al	0 - 900	0 - 900		0 - 50	0 - 500
As			0 - 100		
B	0 - 900	0 - 900		0 - 1,000	
Ba		0 - 5,000			
Bi			0 - 100		
Ca	0 - 3,000	0 - 5,000		0 - 50	0 - 500
Cd		0 - 900			
Ce			0 - 100		
Co			0 - 100		
Cr	0 - 900	0 - 900			0 - 500
Cu	0 - 900	0 - 900		0 - 50	0 - 500
Fe	0 - 900	0 - 900		0 - 50	0 - 500
In			0 - 100		
K		0 - 900		0 - 10,000	0 - 500
Li		0 - 900			0 - 500
Mg		0 - 5,000		0 - 50	0 - 1,500
Mn		0 - 900			0 - 500
Mo	0 - 900	0 - 900		0 - 500	
Na	0 - 3,000	0 - 5,000		0 - 10,000	0 - 100
Ni	0 - 900	0 - 900			0 - 500
P	0 - 3,000	0 - 5,000		0 - 2,500	
Pb	0 - 900	0 - 900		0 - 50	0 - 500
S					
Sb		0 - 100			
Si	0 - 900	0 - 900		0 - 500	0 - 300
Sn	0 - 900	0 - 900			
Ti		0 - 900			
V		0 - 900			0 - 500
W			0 - 100		
Zn	0 - 3,000	0 - 5,000		0 - 50	0 - 500
Zr		Zr	0 - 100		

Table 2: The SpectrOil 100 Series models and calibration ranges in ppm

Advanced Software for Stability and Accuracy

SpectrOil Version 8 software includes recent innovations such as advanced signal processing and background correction capabilities for significant performance improvements. The system provides an intelligent method of measuring peak signals, resulting in a lower LOD by 2x for most elements (Table 3). Active alerts allow a user to know when results could be affected due to instrument drift. New Integrated Standardization is a more efficient workflow that saves time when measuring real samples. Finally, improved instrument self monitoring means better long term stability, so standardization is needed less often and results are consistent over time (Figure 8).

SpectrOil spectrometers provide exceptionally accurate and stable elemental analysis measurements for oils and fuels, making the technique the number one choice for laboratory managers and reliability professionals who require rapid results without sample preparation, gases, glassware or advanced training. SpectrOil analyzers are a natural fit for sites where time sensitive samples are generated and unpredictable sample volumes (frequently five samples, occasionally 100) are common. Consider that Formula 1 Race teams rely on this technology to support their cars, where time sensitivity, reliability, and sub-ppm changes in wear mean the differences between winning and going home (Figure 9).

Laboratories with consistent volumes can avail themselves of the lower cost of sample processing with high accuracy and stability; also automatic sample processing can be justified. RDE spectrometers have always been difficult to automate due to the need to replenish the graphite electrodes after each analysis. The practical solution to RDE spectrometer automation is the SpectrOil M/R D2R2, which employs an integrated autosampler and two graphite disc electrodes (Figure 10).

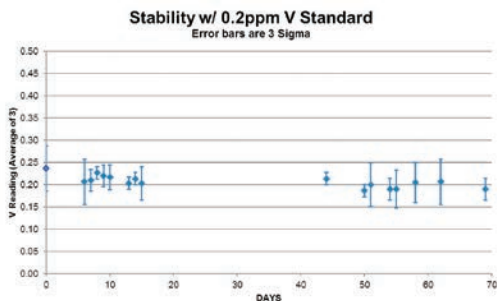


Figure 8: Measurement of a 0.2 ppm Vanadium base oil standard over 70 days at a power generation field laboratory

A robotic arm in the sample changer automatically introduces each of the 48 oil samples in succession, at a rate of 80 samples per hour, without the need for sample dilution.

The entire automation system mounts to the spectrometer sample stand and fulfills all the functions of sequentially introducing and removing oil samples and exchanging graphite electrodes. It is self-contained and works independently of the spectrometer's operating software. Although operation is automatic, it also has the capability to manually sequence through each of the robotics' functions. The newer signal processing and optics hardware make this solution very appealing to commercial laboratories and dedicated support laboratories with consistent volumes.

Element	Typical LOD
Li	≤0.01
Ag	
Cu	
K	
Na	0.05
Cr	
B	
Ti	
Ba	0.1
Zn	
Ca	
Mn	
Ni	
Si	
Al	
Fe	
Cd	0.2
Mg	
V	0.3
Mo	
Sn	1
Pb	
Sb	
P	

Table 3: Typical 2-Sigma LOD (ppm) for SpectrOil calibrated with the CS-24 commercial oil analysis program. The Version 8 software and latest optics technology provide exceptional signal to noise and system performance.



Figure 9: McLaren and Mobil 1 use the SpectrOil 100 Series on the global F1 circuit (Ref: McLaren)



Figure 10: Robotics in SpectrOil M/R spectrometer

CASE STUDY: Railroad Field Laboratory

Railroad and power generation companies with older EMD high horsepower diesel electric engines rely on spectrometric oil analysis to detect abnormal bearing wear in its early stages. These fleets standardize on a low zinc “locomotive oil” to avoid premature wear of bearings and bushings. A commercial oil laboratory placed a SpectrOil in a field lab at a railroad switching yard to support daily maintenance operations. In a summary report of the last three SpectrOil oil analyses Ag (silver) was detected at 2 ppm. This level is a warning alarm, prompting an investigation. In this type of engine, incorrect oil containing a zinc-based additive package can result in severe wear problems. Several components, such as wrist pin bearings, have silver coatings that corrode and wear in the presence of zinc. The early stages of the corrosive action caused by the zinc additive are indicated by the increase in the iron, copper, and silver wear metals. The SpectrOil analysis of other elements – magnesium, phosphorus and zinc helped provide a clue to the root cause of the wear, that the locomotive was topped off with incorrect engine oil. A recommendation was made, based on the analysis, to drain and flush the system, as well as observe correct top-off oil requirements. This particular fault would not have manifested itself this early by any other condition monitoring technique such as vibration analysis, thermography, ultrasound, or performance monitoring. Without oil analysis,

the wear problem could have resulted in a bearing failure and a major overhaul, costing over \$150,000. Nuclear power generation facilities that employ large diesel engines for emergency backup power look for the low detection levels of wear that the SpectrOil can provide also.

In summary, the latest innovations in Optical Emission Spectroscopy, shown in the SpectrOil product line, provide the best elemental analytical performance available today. The new optics and analytical features, combined with the known reliability and stability of the SpectrOil technology make it the ideal solution for elemental analysis in used oil and fuel analysis.



	Fe	Cu	Ag	Mg	P	Zn
30-Sep	19	10	0	0	0	3
23-Dec	21	10	0	0	9	3
23-Mar	27	13	2	107	75	90

Table 4: Spectroscopic results in ppm for an EMD medium speed diesel locomotive. A SpectrOil 110 E system is ideal for railroad field labs.

References

1. ASTM D6595 Standard Method for Determination of Wear Metals and Contaminants in Used Lubricating Oils or Hydraulic Fluids by Rotating Disc Electrode Atomic Emission Spectrometry.
2. ASTM 6728 Standard Test Method for Determination of Contaminants in Gas Turbine and Diesel Fuel by Rotating Disc Electrode Atomic Emission Spectrometry.
3. Rhine, W.E., Saba, C.S., and Kaufman, R.E., “Metal Detection Capabilities of Rotating Disc Emission Spectrometers,” *Lubrication Engineering*, Vol. 42, #12, p 755
4. Lukas, M., and Giering, L.P., “The Effects of Metal Particle Size in the Analysis of Wear Metals using the Rotating Disc Atomic Emission Technique,” presented at the International Symposium on Oil Analysis, Erding, Germany, July 1978.
5. Anderson, D.P. & Lukas, M., “Diesel Engine Coolant Analysis. New Application for Established Instrumentation,” Presented at 1998 Technology Showcase, JOAP International Condition Monitoring Conference, Mobile, AL, April 20-24, 1998.
6. Lukas, M. & Anderson, D.P., *Machine and Lubricant Condition Monitoring for Extended Equipment Lifetimes and Predictive Maintenance at Power Plants*, Proceedings of Power-Gen International, Jakarta, Indonesia, 1996.

3.2: Elemental Analysis: FPQ-XRF for Large Particles

Synopsis

This paper describes a new machine condition measurement system which combines particle count data and elemental analysis information in two closely interlinked measurement phases. This machine condition tool is part of a new portable product that also measures lubricant condition using viscosity and IR to complete the overall condition monitoring picture. The focus of this paper is to describe the new methodologies that apply to the machine condition aspect of the new tool. The paper compares existing analytical techniques used to quantify wear conditions and contrasts them with the new techniques and methodologies the device uses. Finally, this paper presents case studies that use the device to show how the measurements compare to other analytical techniques in different machine condition monitoring applications.

1. Introduction

Particle count and elemental identification answers two of the most important questions in oil analysis: "How many?" and "Where is it coming from?" These two measurements are the most critical in any machine condition monitoring application. Using current technologies, the particle count is often a pre-screen for conducting root cause analysis using SEM/EDX, XRF and in some cases ferrography. These techniques have proven to be expensive, time consuming and very labor intensive. Other routine elemental tests are used but they are particle size sensitive towards the small fines, and they do not offer the best solution for detecting normal to abnormal wear transition.

Machine condition through oil analysis is typically monitored by quantifying the number, size and elemental composition of wear particles produced at the extremities of lubricated machine parts. The size and quantity of these wear particles has a direct correlation to a benign versus an abnormal wear state (Figure 1).

It is important to understand that a benign wear state in one type of machine will be different compared to

another. In these cases, the type of wear mechanism coupled with the contact area, load, speed and lubricant condition all govern the size and quantity of the normal benign wear. This makes limit and alarm settings difficult compared to cleanliness control applications where the overall contamination level must meet a maximum threshold. This threshold is a fixed limit (often specified by the OEM) and it is often small enough to be easily quantified by light blocking laser particle counters. Particle count standards like ISO 4406 and NAS1638 were developed specifically for these applications.

Filtration and other loss mechanisms in lubricant systems, which readily generate wear, also play an important role in the overall particle picture. Filters are primarily responsible for the condition of dynamic equilibrium for a given particle size [1] and set baselines and alarms for large particles. Very fine particles do not work well in this model because they are diluted into the system, making any baseline measurement impossible. The transition from a normal benign wear mode to an abnormal wear mode also creates fewer small particles because the forces acting on the shear mixed layer are now greater, and fine rubbing wear substitutes for much larger wear particles produced from beneath the shear mixed layer [2]. Machines produce different types of wear particles depending on the wear mode. These are explained in greater detail in the Wear Particle Atlas [3].

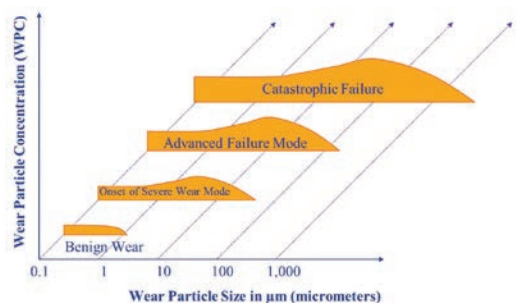


Figure 1: Progressions to Failure

2. Existing Machine Failure Measurement Techniques

Particle Count

Particle count is a good indicator of the severity of a wear situation and the transition from small to large particles can easily be detected. Particle count is usually performed using one of the following techniques: laser light blockage, direct imaging or pore blockage.

Laser light blocking suffers from coincidence effects (particle overlap) and from the ability to see through dark sooted samples. Therefore, this process is limited to clean translucent fluids used in the contamination control industry where internal machine contact is minimal.

Direct imaging counters the coincidence effect by processing particles over a larger area using a CCD sensor. The sample illuminates by a pulsed laser diode which can increase light throughput and overcome dark sooted samples, to about 2% before dilution.

Traditional pore blockage devices are like optical particle counters because they saturate at relatively low levels and are not ideally suited to accurately quantify heavily contaminated machine wear samples. However, they have no difficulty processing oils containing soot or water because these contaminants can pass through the pores without increasing the signal output. This is the primary advantage that pore blockage techniques have over light blocking and direct imaging techniques.

3. LNF Compared to Traditional Ferrography

Atomic Emission Spectroscopy

Elemental identification of wear particles has traditionally been performed using atomic emission spectroscopy by either Rotating Disc Electrode (RDE) or Inductively Coupled Plasma (ICP). Both these techniques are limited when it comes to identifying large particles. As a result, other complementary techniques have been developed to help increase the large particle detection capability of atomic emission. These techniques include Rotrode Filter Spectroscopy (RFS) and acid digestion. These additional techniques are time consuming and require a lot of special sample preparation and, in the case of acid digestion, dangerous chemicals are used.

X-ray Fluorescence (XRF)

XRF is a common technique that quantifies individual chemical elements in used oil samples.

Samples are typically analyzed by taking an x-ray of a small oil sample (1-2 ml) in a cup. Similar to atomic emission techniques, the large particles associated with abnormal failure modes are not suited to the analysis technique using a cup because the focused XRF beam spot does not statistically represent the large particle distribution in only 1-2ml of oil. These results do correlate well with RDE and ICP; however, the overall elemental signal is much lower. Again, this is expected based on the small XRF beam spot compared to the overall oil volume being examined. Interference from small sub micron carbonaceous soot particles also creates issues for heavily sooted diesel engine oil samples using this technique. These types of samples require some form of baseline calibration to compensate for the soot interference.

You can achieve better sensitivity for large wear particles by focusing the beam onto the particulate itself. This is essentially what occurs when you examine particles from magnetic chip detectors using a piece of sticky tape. The RAF early failure detection centers (EFDCs) in the United Kingdom extensively use this technique.

Ferrography and Filter Patch Analysis

Microscopy is a powerful technique for identifying root causes of wear mode and mechanism failures. More advanced ferrography techniques for substrate preparation also identify ferrous from non-ferrous metals and crystalline from non-crystalline materials. Ferrogram analysis is an in depth and conclusive test since it uses heat treatment to identify different types of steel along with particle color, surface, morphology and use of polarized light. The more advanced substrate preparation, such as using a ferrogram maker, differs from straight filter patch analysis in this regard.

The biggest downside to performing ferrography is that it is time consuming and requires an expert to perform the analysis. This skill demands many years of analyzing multiple ferrograms to become skilled in the art. Microscopy techniques need to be coupled with other quicker screening techniques for them to be successful. It is not feasible to run a routine sample history using microscopy alone.

SEM EDX

The SEM EDX technique is used for visually examining particles at very high magnifications and performing spot elemental analysis on the particle using an EDX device. The depth of field is much larger on an SEM compared to conventional metallurgical microscopes. This depth of field enhancement means the complete particle can remain in focus at high magnifications and you can achieve greater detail. Like standard wear particle analysis, using an optical microscope SEM EDX is not suitable for routine sample analysis. The instruments are expensive and the technique involves some sample preparation, such as applying a conductive coating to the sample to help increase resolution. A complete ferrographic analysis. However, if identification of the root cause of the problem is required or further corroboration is needed, we recommend a complete Ferrography analysis.

4. A New Technique – Filtration Particle Quantification Combined with EDXRF

[In this unique system design, machine failure and root cause analysis is interpreted by using a two-step process combining a modified pore blockage technique with an XRF analyzer. Figure 2 shows the tower which encompasses the FPQ and XRF device in the overall oil monitor system. The figure also shows the filter being inserted into the XRF. This relatively quick process can screen out samples with high particle counts and perform a complete 13 element XRF analysis on the resultant sample filter.

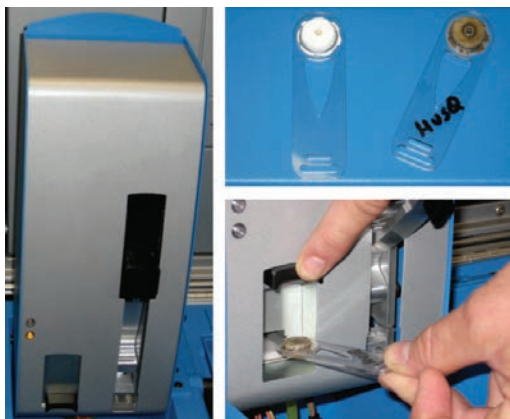


Figure 2: FPQ and XRF tower assembly

Combined Particle Quantifier (FPQ) and XRF Device

The modified pore blockage technique has been termed "Filtration Particle Quantification" or FPQ. The FPQ uses constant flow by driving a 3 ml oil sample using a syringe through a polycarbonate filter with ~ 30,000 4um diameter holes. The resultant pressure drop across the filter, measured with reference to atmospheric pressure is used to quantify particles >4um up to ~1million particles/ml. This is achieved primarily by using a modified filter design compared to a conventional pore blockage instrument. This new patent pending dual dynamic design allows a much greater particle count range (x50) beyond the point where particle swapping and saturation occurs (Figure 3).

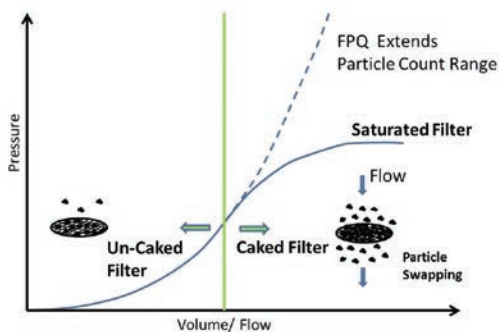


Figure 3: FPQ Filter vs Conventional Pore Blockage Filter

Once the analysis is complete, the filter passes from the FPQ to the XRF device. The FPQ and XRF are closely linked in terms of calibration because of the particle swapping phenomenon. The FPQ and XRF instruments use a series of unique rules and calibrations to ensure accurate elemental quantification of particles up to 1 million particles /ml. This technique combined with the patented filter overcomes the problem with the oil cup analysis which XRF devices typically use. This unique filter design is able to corral the particles into a small area on the filter so the focused X-ray beam can concentrate its energy on those particles. The instrument uses 40kev and 15kev to quantify 13 elements with an average limit of detection of ~ 1ppm.

5. FPQ / XRF Device Case Studies

The case studies that follow demonstrate how the FPQ/ XRF device correlates to existing analytical techniques for measuring particles in various real world applications.

FPQ and X-Ray Correlation to Established Measurement Techniques

The following data set from a series of marine diesel vessels was used to evaluate the FPQ and XRF technology. Samples were analyzed on the FPQ device and XRF and were shown to correlate to LaserNet Fines® and acid digestion using the ICP. A model using an assumed wear particle size aspect ratio and particle mass was used to further correlate the aggregate elemental concentration on the FPQ filter using the LaserNet Fines® and XRF data. Figure 4 and Figure 5 show how the FPQ and XRF correlate to the LaserNet Fines® direct imaging particle counter.

XRF vs. Acid Digestion

LaserNet Fines® direct imaging and spectroscopy are well established techniques to quantify particle count and elemental concentration respectively. RDE and ICP spectrometers lack good sensitivity to detect large particles and they are used as trending tools for fine particles based on a dissolved elemental calibration. An accepted methodology to quantify large particles is to “acid digest” the entire sample by dissolving particles into a liquid which can be quantified using a standard ICP calibration. However, corrosive chemicals, time, cost and effort make acid digestion impractical.

The data in Table 1 shows a selection of marine samples analyzed on the ICP before and after acid digestion. This method is commonly known as differential acid digestion.

Sample	Before Acid Digestion -ICP (ppm)						After Acid Digestion - ICP (ppm)					
	A	B	C	D	E	F	A	B	C	D	E	F
Ag	0	0	0	0	0	0	0	0	0	0	0	0
Al	0	0	0	10	10	21	0	0	0	0	13	28
Cr	6	0	0	0	6	7	6	0	0	0	6	8
Cu	0	0	0	0	11	11	0	0	0	0	10	10
Fe	10	7	0	0	33	67	11	10	0	0	35	86
Mo	0	0	0	0	0	0	0	0	0	0	0	0
Ni	0	0	0	0	0	0	0	0	0	0	0	0
Pb	0	0	0	0	0	0	0	0	0	0	0	0
Sn	0	0	0	0	0	0	0	0	0	0	0	0
Ti	0	0	0	0	0	0	0	0	0	0	0	0
V	0	0	0	0	0	0	0	0	0	0	0	0
Total ppm	16	7	0	10	60	106	17	10	0	0	64	132

Table 1: Differential Acid Digestion Sample Result (Sample E=10-1151, Sample F=10-1149)

Figure 6 shows how the differential ICP results (large particles) for samples E and F compare to the XRF data for the same samples. Note that the XRF data is not shown in Table 1 above. The large particle portion correlates very well (within 3ppm) to the filtered XRF results (Figure 6).

Figure 7 shows the difference in ppm between the ICP and XRF readings for Fe and Al in Sample F. This is an expected result based on how large and small particles behave in a closed loop lubricating system. Large particles get lost and filtered out far more easily compared to fine debris which never gets lost and continues to grow in concentration.

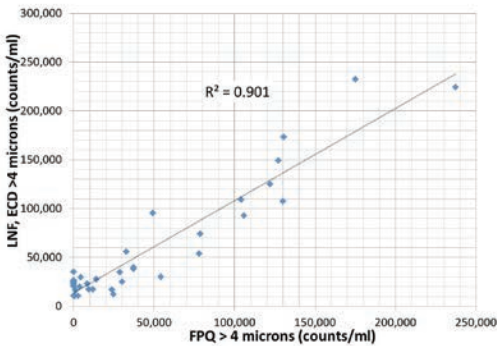


Figure 4: LaserNet Fines® vs. FPQ (counts/ml >4um)

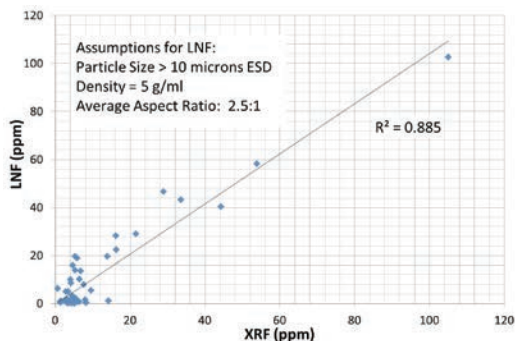


Figure 5: LaserNet Fines® vs. XRF – Total ppm

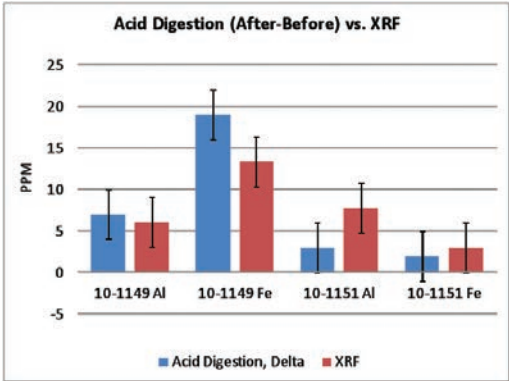


Figure 6: Differential ICP vs XRF (Sample E=10-1151, Sample F=10-1149)

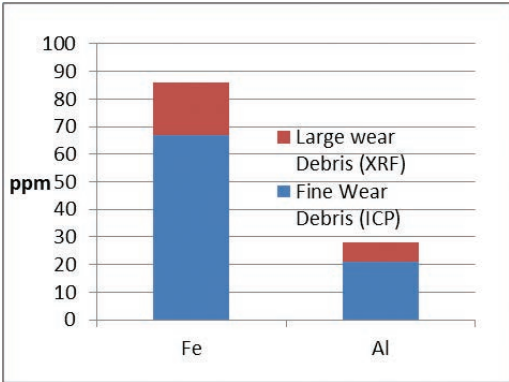
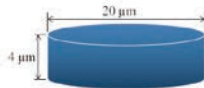


Figure 7: Typical ratio of large to small particles observed between XRF and ICP (Sample F)

PPM (mass) vs Particle Concentration (quantity) on the FPQ Filter

Based on the density of iron, it would take ~100 particles of the illustrated dimensions in 1ml of oil to raise the elemental concentration by just 1ppm. For lighter metals such as Aluminum, it takes approximately three times the amount of particles. This explains why the differential elemental ICP and XRF readings are relatively low when compared to the fine and dissolved particle readings using routine spectroscopy. In this example, the Fe and Al wear particles are most likely caused by cylinder/piston wear. This is a common failure mode in the application and shows how the XRF is able to identify root causes of problems.



Wear Progression to Failure

When a machine enters an abnormal wear mode there is always an increase in the size and production of severe large wear particles. They are identified as an increase from a known equilibrium level in the system. As the abnormal wear progresses, the size and rate of production of these particles increases until the system eventually fails.

Note that fine wear particles detected by RDE spectroscopy and ICP continue to rise in the lube system and are unaffected by filtration or other system loss mechanisms. Take care when changing the oil and subsequently interpreting fine and dissolved wear metal data vs. XRF data. Limits based on rate of change apply in this case. For larger particles measured by FPQ and XRF, a static limit applies after the system reaches equilibrium. This is demonstrated in Figure 8.

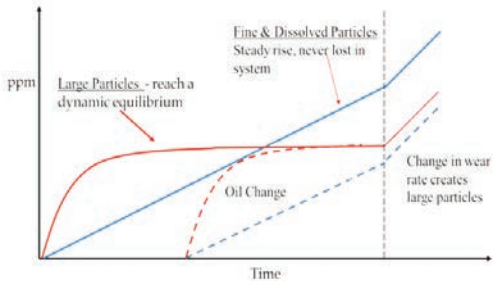


Figure 8: Behavior of large vs fine particles

Unlike existing optical particle counter and pore blockage technologies, the FPQ can handle a wide range of applications with relatively high wear rates (up to 1.0 million p/ml). Table 2 shows FPQ and XRF data for a wide range of components that are typically found in heavy duty industrial vehicle equipment such as engines, transmissions, final drives, and front differentials. The data shows pairs of components with corresponding high and low wear rates.

Sample	Particles >4µm (/ml)		Application	ITS Q5800 XRF (ppm)			
	LaserNet	Fines FPQ data		Al	Cu	Fe	Si
E1 High wear	180209	141795	Engine	2.0	0.0	0.8	1.4
E2 Low Wear	26802	44188	Engine	0.5	0.6	0.6	0.7
T1 High Wear	46618	50390	Transmission	0.4	2.2	2.2	1.7
T2 Low Wear	5346	9664	Transmission	0.0	0.0	0.2	0.3
F1 High wear	213674	226222	Final Drive	4.9	0.0	8.4	7.0
F2 Low Wear	17185	26948	Final Drive	0.1	0.0	2.0	0.5
D1 High Wear	88193	62259	Front Diff	1.2	0.0	4.2	1.9
D2 Low Wear	37613	34773	Front Diff	0.9	0.7	2.9	1.2
E3 High Water	1025329	31686	Engine	0.5	0.0	1.0	0.4

Table 2: Normal and abnormal FPQ & XRF data for various applications

As expected, the particle count on the FPQ correlates well with direct imaging particle counting (Figure 9). In addition, the elemental XRF readings can differentiate between low wearing systems and more critical high wearing systems. This data shows that it is possible to make a recommendation on the root cause of the increased wear rates based on a material map of the lube system.

This data set also demonstrates a unique advantage that the FPQ has when analyzing emulsions and other sample types that contain “phantom” particles included in the overall particle count. Water and other liquids pass through the polycarbonate filter pores and the results are unaffected. Sample E3 contains a significant amount of free water ingestion that produced a highly elevated particle count reading on the LaserNet Fines®. The real particle count in this sample was only ~ 31k p/ml and the elemental level was low.

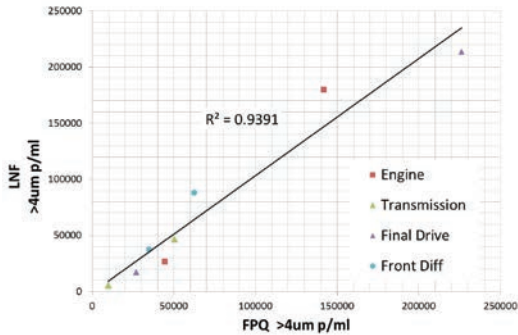


Figure 9: FPQ vs LaserNet Fines®, normal and abnormal wear in different applications

6. Conclusion

The FPQ, with its patent pending dual dynamic filtration system, handles a wide range of lubricant applications with varying wear levels. The particle count using the FPQ filter correlates with existing direct imaging particle counting. The subsequent elemental concentration from the FPQ filter using XRF analysis correlates well with ICP differential acid digestion, demonstrating that the methodology is valid. The combined particle count and elemental concentration identifies changing wear rates and isolates potential root causes of problems in lube systems. Particle count and elemental concentration provides the real elemental break down of particles captured and quantified on the filter. This methodology eliminates many of the problems associated with other techniques such as particle size detection and the impervious nature of many used oils found in heavy duty industrial applications.

References

1. Daniel P. Anderson and Richard D. Driver. “Equilibrium particle concentration in engine oil” *Wear*, Volume 56, Issue 2, October 1979, Pages 415-419
2. A.A., Bowen, E.R., and Westcott, V.C. “Characteristics of Particles Generated at the interface Between Sliding Steel Surfaces” *Wear*, Volume 34 (1975) Pages 261-273
3. Anderson, D.P., “Wear Particle Atlas (Revised)” prepared for the Naval Air Engineering Center, Lakehurst, NJ 08733, 28 June 1982, Report NAEC-92-163, Approved for Public Release; Distribution Unlimited – Pages 125-134

Chapter 4: Particle Analysis

The analysis of particulates in oils, including the amount as a function of size and the type of particulate and its morphology, encompasses the most direct means of any class of instrumentation with which to assess machinery condition and contamination. When a machine enters an abnormal wear mode, the size and population of wear particles increases. In the case of a diesel engine, large severe sliding wear particles are produced when the wear surface stresses become excessive due to load and/or speed. Spectro Scientific's unique collection of particulate analysis tools, headlined by the LaserNet Fines® (LNF) series, widens the scope of complete particulate analysis. It enables extension of the analysis toolbox to encompass systems from aerospace hydraulic systems, which may generate particulates measured in the single particles per 100 milliliter of oil to over-the-road truck engines, which may have millions of particles in a single milliliter of oil. Particular emphasis is placed on providing information in addition to size-based counting, while ensuring that size-based counting is extremely accurate.

Comprehensive particle analysis includes an assessment of the morphology of generated particulates, as provided in ferrography and LNF. Established by over 40 years of research in machinery breakdown, particle morphology can directly pinpoint the type of wear (wear mode) and

thus provide a gauge of the severity of the component wear, while helping to pinpoint the generation source, including external contamination. Finally, the most basic classification in particle analysis, whether the particles are due to wear or external contamination, can be determined by measuring ferrous debris particulates in the oil.



4.1: Dynamic Equilibrium – Large Wear Particles in Oil Lubricated Systems

Several approaches are used to model a machine's wear formation. Wear particles of various sizes reach their own dynamic equilibrium condition based on a balance between production rate and loss rate of particles in a machine. The result of this dynamic equilibrium model is the ability to predict, based on particle count trending information, what is a normal level and the level increase in particle production during the trend towards equilibrium, including at equilibrium, and what is abnormal for a given machine.

We assume that particle production follows a unique behavior per machine and is measured by the analytical instrumentation. For loss rate, during passage of oil through the system (Figure 1) it is assumed that there are various competing mechanisms for the removal of particles, namely:

- 1) Filtration
- 2) Settling
- 3) Impaction and adhesion
- 4) Comminution (grinding up of particles)
- 5) Dissolution (oxidation or other chemical attack)
- 6) Magnetic separation

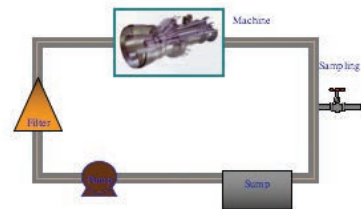


Figure 1: Simplified Machinery Oil Path.

In particular, the rate of increase in concentration of particles in a given size range is:

$$\frac{dC_L}{dt} = \frac{P_L - kC_L}{V}$$

Where:

C_L = Concentration of particles (mg/L)

P_L = Production rate of particles (mg/h)

k = Removal Rate Constant (L/h)

V = Oil Volume (Liters)

t = time (h)

$P_L - kC_L$ = net rate of addition of particles to the oil (mg/h)

Integrating from $t=0$ and $C_L = 0$, and assuming a constant production rate gives us the concentration of in a given size regime as a function of time:

$$C_L = P_L/k(1 - \exp[-(k/V)t]) \quad [1]$$

Once equilibrium is reached, the concentration reaches a steady value of P_L/k . If we assume that the only capture mechanism at work is filtration, and a capture efficiency of 1% for 4-10 micron particles given a 40 micron pleated filter cartridge and a constant flow rate of 7.5 liters/ min (450 liters/h) through the filter we can calculate the Removal Rate Constant for these particles as:

$$k = F \cdot \text{eff} = 450 \cdot 0.01 = 4.5 \text{ L/h} \quad [2]$$

Where F is the flow rate (liters/hr) and eff is the efficiency of the filter (0.01).

We can then immediately calculate the time to reach equilibrium for a given oil volume (Table 1, Figure 2). Defining equilibrium as the point at which the concentration of these particles reaches 99% of its equilibrium level, we have, from Equations [1,2]:

$$t_{eq} = -(V/k) \cdot \ln(0.01) = -(V/F \cdot \text{eff}) \cdot \ln(0.01) = 1.02 \cdot V \text{ hours} \quad [3]$$

The number of times that the oil has circulated before reaching equilibrium can be calculated as:

$$\#Cycles = t_{eq} \cdot F/V = 1.02 \cdot F \quad [4]$$

Note that this is independent of volume.

1% Capture Efficiency (4-10 micron particles)				
Oil Volume Liters	Flow Rate L/h	# Cycles	Total Volume (L) to equilibrium	Time to reach equilibrium (hours)
40	450	459	18360	40.8
20	450	459	9180	20.4
10	450	459	4590	10.2
5	450	459	2295	5.1

Table 1: Time to equilibrium for different oil volumes assuming a 1% capture efficiency and 7.5 liter / min filter flow rate.

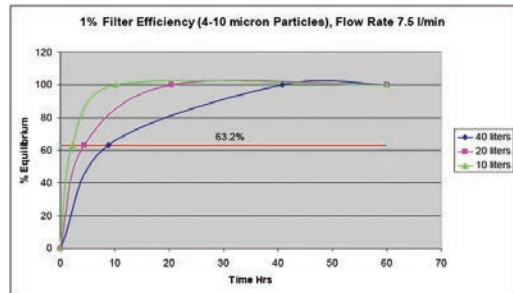


Figure 2: Time to equilibrium for 10, 20, and 40 liter sump capacities.

This simple diesel engine example shows that oil volume capacity, oil flow rate, filtration efficiency and particle size will all have an effect on the time to equilibrium. The time to equilibrium is critical to the oil analyst because it governs when a sample should be taken either between an engine overhaul or routine oil change. This example demonstrates that the time to equilibrium is relatively quick even for the biggest capacity engine. However, this may take much longer in other applications where there are multiple loss and production mechanisms.

4.2: Ferrography – Particle Size and Morphology Analysis

The ferrography technique was developed in the 1970s to overcome the large particle detection deficiencies of spectrometric oil analysis. It is a technique used for the separation of particles from fluids for microscopic examination and subsequent analysis. The name ferrography derives from the initial development of the methods to precipitate ferrous wear particles from engine lubricating oil. The original analytical ferrograph used a strong magnetic field gradient to separate wear particles from the used oil sample for subsequent microscope examination.

Since that time, a number of different instruments have been introduced such as the rotary particle depositor which also separates wear particles for microscopic examination along with other instruments which quantify the amount of ferrous wear debris in an oil sample. The primary instrument in ferrography is the analytical ferrograph which is used to prepare a ferrogram. A non-wetting barrier is painted on one surface of a microscope slide. This coating traps the fluid delivered by a peristaltic pump. In use, the slide is mounted at a slight horizontal angle, permitting the fluid to flow by gravity along the glass but within the barrier, where it finally is picked up by a drain tube.

The slide is mounted above two permanent magnets which are separated by an aluminum sheet. The poles of the magnets are counter posed. That is, where one magnet pole is considered north, the pole of the other magnet across the aluminum strip is south. Positioning the magnets in this way causes a strong magnetic field gradient to be created in the vertical direction above the aluminum strip. Magnetic particles in the fluid experience a strong downward force. These particles migrate through the fluid down to the glass surface, where they are deposited in strings perpendicular to the direction of fluid flow (Figure 1).

After all the fluid in a given sample has run across the slide, a fixer solution is passed over the slide to remove residual fluid. When the fixer has evaporated, the slide is ready for observation using the microscope. Ferrous particles are deposited on the slide according to size. The force acting on a particle is proportional to volume, but the viscous resistance of the suspending fluid is proportional to surface area. Therefore, for spheres force increases with the cube of the diameter (volume) but resistance increases only with the square of the diameter (contact surface) [1].

The largest ferrous particles, therefore, are deposited at the entry region of the slide where the lubricating oil first touches down on the glass surface. At a position

farther along the slide, all ferrous particles larger than a characteristic size already will have been precipitated. For non-ferrous particles, such as aluminum, brass, white metal, etc., precipitation will often occur because these materials are weakly magnetic. However, the deposition of these materials will be less size-selective. Consequently, large particles of nonferrous metal may be found anywhere along the length of the slide as also shown in Figure 1.

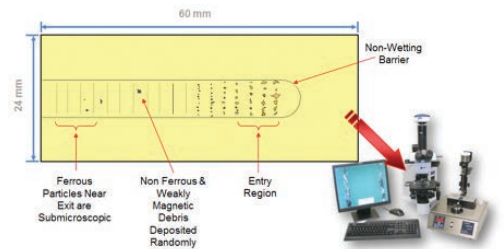


Figure 1: Example of a Ferrogram. On the lower right is an image of the Spectro Scientific Thistle Tube Ferrography Lab including ferrography slide maker, microscope and wear particle atlas (WPA) software with reference pictures.

Examination of the slide in a bichromatic microscope reveals details of size, shape, and number of particles. From this information the condition of oil-lubricated parts may be assessed [2]. Machines operating normally usually generate small flat particles at a slow steady rate. If the number of particles increases, and particularly if the ratio of large to small particles increases, we have an indication that a more severe mode of wear has begun. The generation of large severe wear particles signals the imminent failure of the wearing surface.

Different wear mechanisms each generate characteristic particles. Six regimes of wear have been identified with sliding wear. Miscellaneous shaped particles are associated with break-in wear. Small platelets are associated with normal rubbing wear. As operating parameters become more arduous, the metallic rubbing wear particles increase in size and become oxidized. Finally, just prior to failure, large, metallic, severe wear particles are produced.

Abrasive wear, analogous to a crude machining process, generates particles in the form of loops, spirals, and bent wires. Increase in the number and size of these particles shows that an abrasive wear mechanism is progressing rapidly.

In addition, two types of particles are associated with rolling mechanisms. These are fatigue chunks and laminar particles. Fatigue chunks represent material removed as rolling elements spall. Laminar particles, which are large, thin plates, result from material being passed through the rolling contact. Combined rolling and sliding, as in gears, produces scuffing particles and fatigue chunks. Determination of the composition of particles can establish their origin. The site of deposition, reflectivity, and color of particles aid in their identification. Figure 2 shows examples of abrasive wear, sliding wear and fatigue wear.

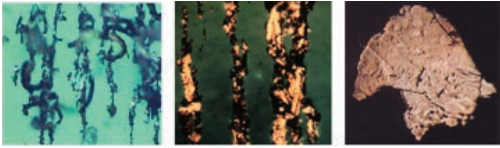


Figure 2: Example of cutting wear, severe sliding wear and fatigue wear

Ferrography can be useful to detect situations that are not obvious, such as water in oil as described below:

Used oil samples were taken from a 1200-kW, turbine-driven reduction gearbox that had just been overhauled. A wear particle baseline was established after five days of operation. No severe oxides or crystalline particles appeared on the ferrograms at that time. The only reservation the analyst had was that a certain number

of dark metal oxides were found at the entry deposit. The oil sample taken after one month of operation, however, reflected a greatly deteriorated wear situation. Many red oxides, as well as free metal particles with tortured and oxidized surfaces, were found. Water in the lubricant not only caused an oxidative attack but the load carrying ability of the lubricant was reduced, resulting in large, abnormal wear particles. As a result of the tests, oil-water separators were installed in the lubricating system and follow-up sample analysis returned to normal.

Ferrography techniques are commonly used in oil analysis laboratories. Ferrography provides comprehensive information about machine wear; however, the technique is fairly complicated and requires a specially trained technician to perform the test. In industrial plants, wear debris analysis (WDA) is more commonly used as it is fairly easy to prepare a sample. Oil is passed through a filter media and the filter is later examined under a microscope. Size, shape and morphology on large particles are examined carefully and compared with reference pictures in the library. It is a good entry level wear debris analysis option that and still being offered as part of Spectro Scientific's MiniLab Series Analyzer.

References

1. Anderson, D. P.: Analysis of Gear Wear through Ferrography, Equipment Management. (July 1983), 51-56.
2. Bowen, E. R.; Westcott, V. C.: Wear Particle Atlas, Prepared for Naval Air Engineering Center, Lakehurst, New Jersey under contract N. N00156-74-C01682, (July 1976).

4.3: Particle Count

Particle counting is a critical aspect of any machine conditioning program. There are many tools available to monitor and track the quantity and severity of the contamination, whether it comes from external contamination or machine wear. The specific application and type of particles will often determine the best particle counting technique to use. The continuous cleanliness of a hydraulic system is critical and even very low levels of dirt ingress can clog actuators and valves, leading to premature failure. Conversely, gear and transmission systems with lots of moving parts are able to tolerate many more wear particles than a clean hydraulic system.

ISO cleanliness code represents the cleanliness of the oil. Each ISO code represents a range of particles per ml of fluid. Table 1 shows common ISO codes and their corresponding particle count ranges.

ISO Code	Lower range #/ml	upper range #/ml
24	80000	160000
23	40000	80000
22	20000	40000
21	10000	20000
20	5000	10000
19	2500	5000
18	1300	2500
17	640	1300
16	320	640
15	160	320
14	80	160
13	40	80
12	20	40
11	10	20
10	5	10
9	2.5	5
8	1.3	2.5
7	0.64	1.3
6	0.32	0.64

Table 1: ISO cleanliness codes and their corresponding particle count ranges

ISO 4406 reports three cleanliness codes from each particle counting test. Each code represents the number of particles per ml for particle size larger than 4, 6, and 14 microns.

Common particle counting techniques for in-service oil analysis are light blocking, pore blockage, and laser imaging (LaserNet Fines, or LNF).

Light blocking particle counters

Laser light blocking particle counters, or optical particle counters (OPC's) are the traditional instruments used for in-service oil analysis. The working principle of traditional light blockage particle counters is depicted below. A light source, typically a laser, passes through a sample. The light is partially blocked by particles, so less light reaches the photodetector array, resulting in a change in voltage proportional to the area of the particles. The photo detector technology is the same principle used in garage door openers.

Traditional light blockage particle counters have several inherent design limitations. The photo detector results contain measurement errors caused by the presence of water and air bubbles within the oil sample. Properly preparing your sample by using ultrasonic agitation helps reduce the impact of air bubbles on particle count. For water containing samples (an oil sample that is 'milky' contains water), it is common to need 'water stripping' solvents to get a more accurate count. The presence of water results in a significant error in the reported particle count.

The advantage of the light blocking particle counting

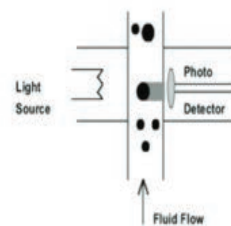


Figure 1: working principle of light blocking particle count technology

technology is that due to its simple principle of operation, it can be deployed in laboratory instrumentation, portable devices, or on line sensors and they all share the common platform and provide similar

results. The Spectro Scientific MicroLab 40 automated oil analysis system utilizes light blocking technology for particle counting.

The disadvantages of this technology are that it is fairly easily to get “fooled” by water droplets or other “soft” particles such as silicon based anti-foaming additives in oil. They will be counted as particles and the oil may appear dirtier than it actually is. If the oil is dark with soot, this technology does not work. That is one of the reasons why particle count is often not provided in a commercial laboratory report. Another commonly cited constraint of light blocking particle counters is the dynamic range. When particles in oil pass through the small opening in the particle counter, the probability of two or more particles overlapping and appearing to be one particle increases significantly as the number of particles per ml of oil increases. This error is called coincident error. Coincident error dictates the upper range of a light blocking particle counter without dilution of the oil.

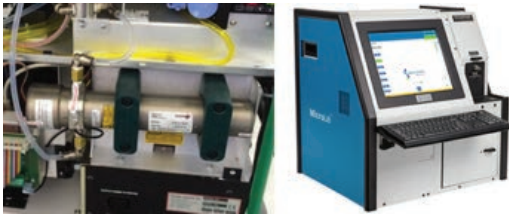


Figure 2: Light blocking particle counter used in the Spectro Scientific MicroLab 40 automated oil analysis system

Pore blockage

Pore blockage particle counters are used as on-site particle counters for in-service machinery oils. They employ a fine mesh whereby particulate accumulates on the mesh. These particle counters are based upon either a constant flow or constant pressure design. Constant flow instruments measure the pressure drop across the mesh while holding flow constant. The constant pressure designs measure the change in flow rate while holding the pressure constant. As particles larger than the pore size deposit on the mesh, they increase the resistance of the flow (figure 3). So for a constant flow system, the pressure will increase over time. While for a constant

pressure system, the flow will decrease over time. The change rate of the flow resistance is a function of number of particles larger than the pore size of the mesh in the oil. The more particles there are in the oil, the faster the flow resistance changes.

In both cases, the particle count distribution is estimated by extrapolation. A typical pore blockage mesh design yields one or two ISO codes. Pore blockage particle counters are rarely used by commercial laboratories due to the limited data generated but can be of great use where interference from water, soot or additives is highly prevalent in the samples.

The advantage of the pore blockage particle counter is its tolerance to oil samples. It will give fairly good result regardless of water, soot, or other “soft” particles in the oil. Also sample preparation is a lot easier. Air bubbles generated during the shaking and agitation process will not affect the test results. So it is a very good technique for portable or on site applications. Pore blockage was selected for particle counting in the Spectro Scientific Fieldlab Q5800 Expeditionary Oil Analysis System. After the particles from the sample are trapped by the filter, they can be further analyzed for elemental composition using XRF.(Figure 3).



Figure 3: Pore blockage particle counter used in the Q5800 Expeditionary Fluid Analysis System

4.4: Ferrous Particle Analysis

Historically, one of the most cost-effective indicators of machinery health has been the analysis of ferrous debris in oil. Such analysis utilizes the ferromagnetic properties of generated debris to perform the analysis. Since nearly all wear debris contains ferrous material (in particular iron), an abnormal increase in the amount of debris can be directly correlated to abnormal machinery conditions in the vast majority of cases. One type of ferrous debris analyzer is the magnetometer. The device works by sensing the disruption of a magnetic field that is generated due to the presence of magnetic particulates in the oil. Since the ferromagnetic properties of iron are well-known, the amount of disruption may be directly quantified to a certain amount (ppm) of iron. Devices such as the FerroCheck, shown in Figure 2, operate on this principle.



Figure 2: The FerroCheck ferrous debris analyzer. Operators use a touchscreen and receive answers in seconds.

The device has several advantages relative to other means of debris analysis. By its very nature the Ferrocheck is sensitive at the single ppm level to ferrous debris, it requires no sample preparation of the 1.5 ml sample size of oil and it can provide answers in seconds. Since the magnetic field generated by the device is so small, the FerroCheck is portable and can operate off an embedded battery without further preparation. To simplify operator sample handling, the FerroCheck analyzes oil in a disposable plastic syringe that may be directly obtained from a standard oil bottle. Upon measurement completion in ~30 seconds, the syringe may be disposed and the oil may be returned to the bottle if necessary. Note that with this mode of measurement, the FerroCheck may also be utilized for the analysis of greases.

Magnetometers for oil analysis come in two general flavors: Those tuned to the detection of single particles in a flow stream of oil and those for the detection of the total amount of ferrous debris in a static sampling

of fluid. Both utilize the same principle of measurement but are typically tuned to the particular mode of measurement by (a) the geometry of the oil sample when analyzed, and (b) the signal processing of the magnetic field disruption. It should be noted, as in the case of the LaserNet 230, that these two general measurement modes may be mixed. In the case of the FerroCheck, the geometry is optimized to provide single ppm sensitivity with the minimum amount of fluid. When the syringe drops into the FerroCheck measurement chamber, the disruption of the magnetic field is immediate, as shown in Figure 3. The FerroCheck is phase-sensitive and so it can distinguish between disruptions in the magnetic field due to ferromagnetic particulates and disruptions due to inductive currents generated by highly conducting samples (such as aluminum). The amount of ferromagnetic disruption is then immediately related to the ppm of total ferrous material. This total amount is not sensitive to size distribution of the particulate but rather only the total mass of magnetic material, which is assumed to be primarily iron.

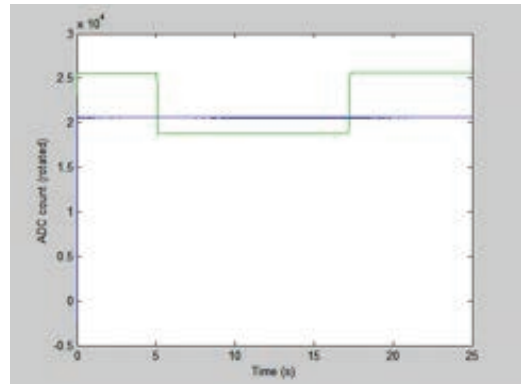


Figure 3: FerroCheck response to a 500 ppm sample as it is inserted at 5 seconds and removed at 17 seconds. The green trace indicates the response due to ferrous material and the blue due to electrically conductive material. The FerroCheck uses this phase-sensitive detection to quantify the amount of ferrous material. This is accomplished by relating the signal drop in the green channel to the ppm of ferrous material. By the nature of the measurement, the response is linear over the entire range of measurement.

The FerroCheck can sense the total amount of debris between 0-2000 ppm of material. Again by the nature of this magnetometer-based device, the accuracy of the measurement remains high across the measurement range, as illustrated in Figure 4. This is counter to the case in elemental analysis systems, where more material

generally leads to a more complex signature to analyze. In fact, the range of the measurement is only limited by the sensitivity of measurement needed and the range limits of the analog-to-digital converter utilized. These systems can be linear up to percents of ferrous material in the oil.

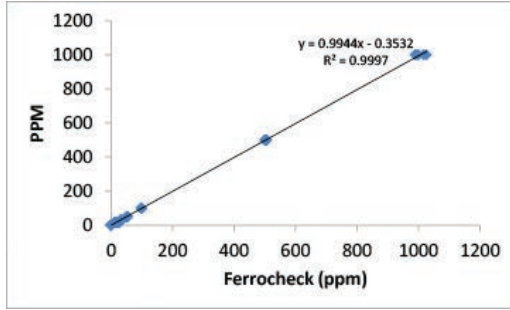


Figure 4: FerroCheck-determined ppm versus known ppm of magnetic material, with repeated measurements at each concentration (5 measurements at all levels save for the 15 ppm sample, which is measured 20 times in order to determine the detection limits of the device). It can be seen that the system is highly linear and accurate across the range of measurement.

The particular magnetic design and signal processing approach of the FerroCheck is tailored to provide a device with readout which is insensitive to temperature over its range of operation. Similar to the LaserNet 230, the device utilizes four coil windings on two coils, two each for sense and driving (generation) of the magnetic field. The drive windings are wired in series so that the identical current flows through both coils, while the sense windings are wired in anti-series so that with no sample the output of this magnetometer is essentially

=0. The syringe is inserted into one coil only, and so the actual signal generated is always the difference between two identical coils, one with the oil and one no oil. This greatly stabilizes the measurement and enables repeatable ppm-level detection.

For processing the output of the FerroCheck into a ppm reading, the device uses the response of both the physical insertion of the syringe (via position sensors) and the electromagnetic response (the signal from the magnetometer itself). This allows for accurate determination of the time at which the oil enters and exits the measurement chamber. Further, since the two events (insertion/removal) are both monitored, a double measurement of the amount of ferrous material is made each time the sample is inserted and removed. This redundancy further enhances the accuracy and error-checking capabilities of the FerroCheck. Since in both cases (insertion/removal) the electromagnetic response is instantaneous, a third response, that of the non-magnetic channel of the FerroCheck, may be utilized to sense and correct any temperature-induced variations in the signal at the time of measurement in the magnetic channel, since variations in the signal outside the insertion and removal times is temperature-related. This is particularly important if the sample is fresh off machinery and is hot, for example.

The FerroCheck is a unique device that takes advantage of the extreme simplicity and sensitivity of coil-based magnetometer systems, while at the same time building in ancillary sensing and advanced signal processing methodologies to ensure the raw response of such a system is stable to environmental effects. In addition to an intuitive, large touchscreen interface and simple navigation, the device is ideal for operators who are new to the technology with varying levels of experience.

4.5: LaserNet Fines® – Particle Count, Particle Shape Analysis and Ferrous Particle Analysis

This paper describes how using the state-of-the-art LaserNet Fines® (LNF) Q200 Series instruments in your lab provides an accurate, cost-effective solution to traditional methods of analyzing oil debris particles.

LNF offers a “one-stop shopping solution” to identifying the type, rate of production, and severity of mechanical faults by measuring the size, distribution, rate of production, and shape features (“silhouettes”) of wear debris in lubricating oil. The recent addition to the LNF series, LNF Q230, adds ferrous particle analysis

capability. This makes LNF Q230 the most advanced and comprehensive wear particle analyzer on the market. No longer are highly trained human analysts and time-consuming calibration procedures at specified flow rates required to obtain accurate particle counts and particle shape classification.

The focus of this paper is to provide details on how the LNF operates and how its method of particle counting and optical image analysis compares to laser style particle counters and traditional analytical ferrography.

Finally, two case studies, one for an Engine Test Cell and one for a Gearbox Accelerated Failure test, are discussed in the paper to demonstrate how the LNF technology offers unique active machine wear results, without the qualitative subjectivity and potential cost of traditional ferrographic analysis.

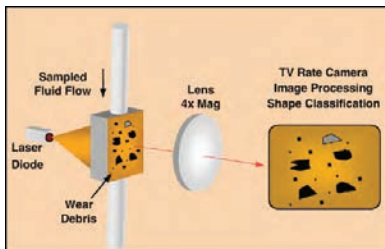
Introduction – LNF Technology

Jointly developed by Lockheed Martin and the Naval Research Laboratory with the Office of Naval Research, LNF is a particle shape classifier that also provides a highly accurate particle count for particles greater than 4 μm using laser imaging techniques and advanced image processing software. Silhouette images of all particles larger than 20 μm in major dimension are automatically classified into the following categories:

- Cutting
- Severe sliding
- Fatigue
- Nonmetallic
- Fibers
- Water droplets

The instrument counts these particles and provides a quantitative measure of active machine wear. Bitmap images are saved and printed on report for review. Reliability engineers can make more informed decisions using LNF data by trending both the total particle size distribution and the sub category particles. In addition to solid particles, the percent of free water is estimated based on the calculated volume of the detected water droplets greater than 20 μm while air bubbles greater than 20 μm are recognized and eliminated from the count. The instrument automatically corrects for the color of the fluid, making it accurate for intrinsically light and dark-colored fluids such as in service engine oils. The basic operating principle of LNF is illustrated in Figure 4-1 below.

Figure 4-1: LaserNet Fines® Operating Principle



1. A representative oil sample is drawn from the lubricating system and brought to the unit.

2. The oil is drawn through a patented viewing cell that is back-illuminated with a pulsed laser diode to freeze the particle motion.
3. The coherent light is transmitted through the fluid and imaged onto an electronic camera.
4. Each resulting image is analyzed for particles.

For wear particles in lubricating oil, the instrument displays particle size in terms of maximum chord. For particles in hydraulics, it displays the size in equivalent circular diameter for compatibility with ISO cleanliness codes. In either fluid, shape characteristics are calculated for particles greater than 20 μm , and the particle is classified into either a wear category or contaminant category.

Classification is performed with an artificial neural network developed specifically for the LNF system. Shape features were chosen to provide optimal distinction between the assigned classes of fatigue, cutting, severe sliding, non-metallic particles, fibers, water bubbles, and air bubbles (Figure 4-2). An extensive library of particles, which were identified by human experts, was used to train the artificial neural network.

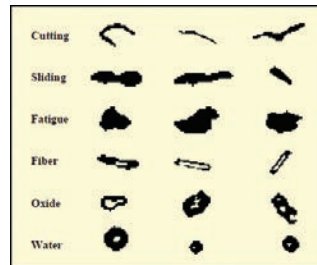


Figure 4-2: Examples of Particle Class Morphology

LNF Compared to Optical Particle Counters

LNF performs the same function of laser particle counters and also performs analytical ferrography tasks. LNF uses a two-dimensional sensing array (640X480 pixels) while the particle counter uses a one-dimensional array. This added spatial diversity allows LNF to examine much higher particle concentrations without special sample preparation in addition to extracting particle morphology. With the ability to recognize shape, the particle counts of LNF are not contaminated by the presence of air bubbles or free water. Instead, those items are both subtracted from the debris counts, with the free water fraction identified separately. The remaining wear debris and filter fibers are included in the total counts and also display in their own distributions.

There are three primary problems with the method existing laser-based particle counters use to determine the size of particles:

- First, they use a point source detector instead of the LNF instrument's two-dimensional detector. It is like counting cars on the side of a multi-lane highway. Overlapping particles will be counted as one bigger particle and skew the final distribution.
- Second, the particle counters must be calibrated and used at a specified flow rate. The accuracy of the detection channels rely on a known flow rate for proper counting and for determining the total sample volume. The LNF detector is highly immune to flow rate variation because it freezes particle motion with a short laser pulse. Sample volume is known from the fixed dimensions of the viewed volume and the number of frames processed.
- Last, traditional obscuring laser particle counters misrepresent the size of non-metallic particles (e.g. silica, dust) because these particles can appear to have translucent centers (see Figure 4-2 above) at the wavelengths the laser uses. LNF uses image processing to "fill-in" the translucent centers before calculating the particle's equivalent circular diameter, therefore accurately reporting the size of oxides and other semi-translucent dust without special calibration.

LNF does not require calibration with a Standard Reference Material because the measurement accuracy is intrinsic to its configuration. Its particle size measurements rely on the camera's pixel size and the magnification power of the optics – both are fixed elements which remain virtually unchanged over time. The measurement volume relies on those fixed elements and also on the thickness of the viewing cell, which is also fixed and does not significantly change with time or operating temperature.

Table 4-1 below compares the Particle Counter with LNF according to various analysis factors.

The most common question regarding the LNF is: "How well does LNF correlate with laser-based, optical particle counters (OPCs) on the market?"

If the samples are properly prepared, LNF particle counting results will correlate to conventional particle counters, with the following notes:

- LNF can count heavily contaminated samples (>5M particles/ml) without dilution, while for conventional particle counters it is very difficult to count such dirty samples (the samples have to be very heavily diluted).
- The upper size limit for LNF is 100µm (the flow cell path as well as the pore size of the screen filter). However, laminar flow coupled with the large aspect ratio of some particles means that particles >100µm can also be reported, such as for fibers and hair while in some legacy reports, counts of particles of 250µm were reported using conventional particle counters.
- When interfering soft particles are present in the oil (such as water droplets, silicone particles from anti-forming additives), correlation between LNF and conventional particle counters is difficult
- Interference from soot up to 2% without the need for dilution.

LNF Compared to Traditional Ferrography

Ferrography has long been the standard method for determining the type of wear mechanisms and severity of faults in lubricated machinery. Here are four primary drawbacks of traditional ferrography:

- First, the test is time consuming. Because of this, Ferrography is often performed on a case-by-case basis. Results from other tests like RDE and LNF are used to screen for a Ferrography test.
- Second, to achieve meaningful results a trained analyst is required.
- Third, the ultimate result is strictly qualitative. Each analyst has their own methodology and preferences for analyzing a prepared slide. Even though most oil analysis labs diligently train their analysts to think the same, inconsistencies are still present and even more obvious from lab to lab.

This is where LNF bridges the gap, providing sample to sample screening insight into possible wear mechanisms and fault severity in a fraction of the time and without the need for a highly trained analyst. LNF counts and classifies all particles in its viewing cell to provide quantitative, repeatable measurements useful for trending and the early assessment of machine condition. Table 4-3 on the right compares Analytical Ferrography with LNF according to various analysis factors.

Using Ferrography as a Follow-up Analysis Technique

LNF analyzes the outline shapes of particles, or their “silhouettes.” Because the optical system within LNF uses transmitted light (back lighting), it is not possible for LNF to distinguish particle color, texture or surface attributes. These are extremely important attributes to consider when making an important root cause diagnosis. Therefore, the results you obtain for each wear category are only typical of that type of particle when it is viewed as a silhouette.

We recommend that if the size or quantity of particles in one of the abnormal wear particle categories (such as severe, fatigue or cutting) increases over a period of time, perform a microscopic examination to validate the particle classifications made by LNF. Ferrography (traditional Ferrography or Rotary Particle Deposition) or membrane filtration (filtergram) are possible follow-up techniques. Other types of non-machine wear related particles such as molybdenum disulfide; carbon flakes and seal material will be classified in one of the severe, fatigue or cutting wear categories depending upon their shape. This is because these particles block light and present a solid silhouette that the shape-recognition software categorizes as one of the solid particle types, that is, as sliding, fatigue or cutting.

	Analysis Time (Volume)	Flush Time	Coincidence Limit	Soot/Opacity	Free Water	Filter Fibers	Calibration
Particle Counter	1.5 min (~20ml)	0.5 min	<90x10 ³ p/ml	Skews Counts	Skews Counts	Skews Counts	To Selected Standard (6 months)
Lasernet Fines	2.3 min (.65ml)	1.5 min	>1x10 ⁶ p/ml	Auto Baselines	IDs Separate	IDs Separate	Intrinsic (Not Required)

Table 4-1: LNF Compared to Laser Style Particle Counters

Consideration Factors	Optical Particle Counters (OPC)	LaserNet Fines	Correlation
Calibration standards	Each OPC is calibrated with a specific particle standard with known distribution before use.	LNF does not require calibration standards to “calibrate” as the optics and CCD ensure a fixed resolution on particle size.	Usually correlates well.
Dilution	Dilution is needed for dark oil, dirty oil or high viscosity oil.	Dilution needed for high viscosity oil.	Dilution factor applies to both LNF and OPC. However, LNF can work fine with most samples without dilution.
Coincident effect	Coincident effect (overlap of particles) in dirty oil causes counting errors for OPC.	No Coincident Effect with flow and cell and direct imaging.	OPC with over count larger particles and under count smaller particles.
Saturation	OPC signal will saturate when particle counts exceed upper limits (30k to 399K/ml).	LNF can handle very dirty oil with 5-10 Million particles/ml.	OPC readings at higher particle counts are not accurate.
Electrical drift	OPC requires annual calibration to correct the electrical drift of detectors.	No field calibration is needed for LNF.	Depending on the direction of electrical drift, OPC can over count or under count particles in the oil.
Particle drifting	It typically takes 5 to 15 minutes per sample for OPC due to multiple reading needed for averaging. More particle settling.	It takes 2 to 4 minutes for LNF to analyze a sample. Much less particle settling.	Less particle settling means more repeatable results.
Sample preparation	No known good automation system for OPC.	ASP works well with LNF. The total automated solution is very cost competitive.	Automation means more repeatable results, eliminating sample preparation variations.

Table 4-2: Consideration Factors

	Prep Time	Analysis Time	Debris ID	Ferrous/ Nonferrous ID	Free Water	Operator Skill Level	Results
Analytical Ferrography	20 min	0.5 min	<90x10 ³ p/ml	Skews Counts	Skews Counts	Skews Counts	To Selected Standard (6 months)
Lasernet Fines	2.3 min (.65ml)	1.5 min	>1x10 ⁶ p/ml	Auto Baselines	IDs Separate	IDs Separate	Intrinsic (Not Required)

Table 4-3: LNF Compared to Analytical Ferrography

LaserNet Fines® Ferrous Particle Analysis

The LaserNet 230 offers a patent-pending, completely unique, coordinated analysis of particulate via imaging and magnetic sensing. The instrument is the only technology of its kind available in the industry today.

We can characterize BOTH the image of a 20-micron or larger particle AND determine if the particle is magnetic. We can bin the size and compare both the image-based and magnetic particle analysis. The Q230 thus provides a strong measure of the nature of the debris – is the debris primarily magnetic (e.g., machine wear) or not (e.g., contamination via sand ingress)? In that sense, the Q230 provides a first-layer elemental analysis unmatched in any instrument. Beyond these features, the Q230 examines smaller and dissolved particulate via a highly sensitive magnetometer, which provides an overall ppm reading of this particulate in addition to the large particles. With two distinct magnetic sensors onboard the Q230, we can reveal information never possible until now, with quantitative debris analysis for 0-100 micron particles. By integrating this magnetic analysis into the existing LaserNet 200 enclosure and streamlining the process with the magnetic sensing devices in normal imaging cell flow path, we are able to acquire this additional information with processing times of 15 seconds, completely invisible to the operator.

Figure 1 shows an example trace where magnetic particulate is present during an analysis. Each peak, representing an individual magnetic particle, is identified and quantified via a peak detection algorithm embedded in the Q230. The red dots indicate the beginning and end of an identified magnetic particle peak. At a peak threshold of approximately 10 counts and a 20 micron spherical magnetic particle having a signature of 30 counts above the baseline we can clearly see individual magnetic particles all the way down to 20 microns in diameter. During the manufacturing process, the response of each Q230 unit is calibrated to ensure that it provides an accurate and highly sensitive magnetic particle determination of individual magnetic particles. This is validated before shipment of each unit via test with LNF-545 check fluid. The fluid contains a near-monodisperse mixture of 42 micron magnetic particles. Each Q230 unit must correctly determine the average magnetic particle size to within +/-5 microns before shipment.

The second magnetic sensor, which determines overall magnetic ppm in the sample being analyzed, is designed to be highly accurate across the range of environmental conditions that can be encountered by the Q230. During

manufacture, a calibration is performed to ensure that, under any circumstances, the unit must consistently determine the 0 ppm point to within +/- 7 ppm. An eight hour temperature, solvent, and viscosity sweep is performed to ensure this is the case for each unit. The LNF-545 fluid also contains a homogeneous distribution of nearly dissolved, nanometersized magnetic particles with a calibrated total magnetic content of 100 ppm. The final calibration step for any Q230 involves ensuring that this magnetic content is accurately determined to within +/-10%.

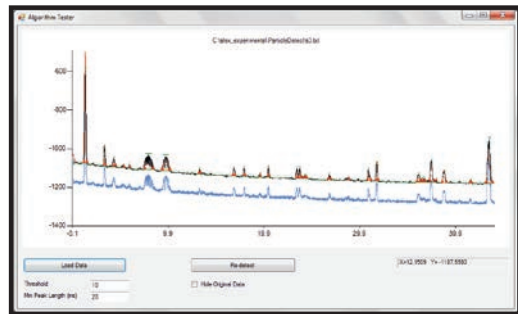


Figure 1: Example raw signal of individual magnetic particles passing through the Q230. The peaks in the data indicate that an individual magnetic particle has passed through the device. The red dots indicate the software's determination of the beginning and end of each peak. The hatches on the top of each peak indicate the Q230's determination of the height of each peak using its built-in peak detection algorithm.

These magnetic signals are determined by comparing the dc signal in the magnetometer, during sample analysis, when there is solvent in the flow line. This is illustrated in Figure 2, which shows an example trace of the response of the magnetometer to a magnetic fluid. The 100 ppm sample produces a raw signal of 770 counts, (i.e., the device outputs approximately eight counts per ppm of magnetic material). Due to the extreme accuracy of this magnetometer subsystem, all Q230 units manufactured have essentially this identical sensitivity. With an acceptable instantaneous noise level during manufacture of 20 counts in the magnetometer, an instantaneous noise equivalent of at least 2.5 ppm is validated upon manufacture in each Q230. As the device averages 2500 samples during its five second measurement time, sub-ppm noise levels are standard. Other application notes, which compare the performance of the magnetometer with industry standards, show the technology considerably outperforms any available on the market.

Figure 3 shows repeatability of the magnetometer performance after 20 consecutive runs. A 3 sigma repeatability of 2.5 ppm out of 33 ppm beats the best performing magnetometers in the industry by several folds.

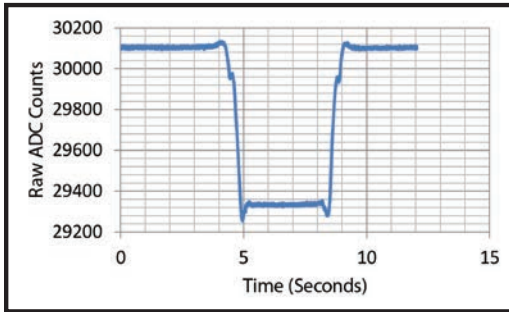


Figure 2: Example magnetometer trace between when the cell contains a 100 ppm magnetic fluid (between approximately 5 - 8.5 seconds) indicated by the dip in the signal and when no magnetic fluid is in the cell. This shows the extreme sensitivity of the device to the presence of magnetic fluid.

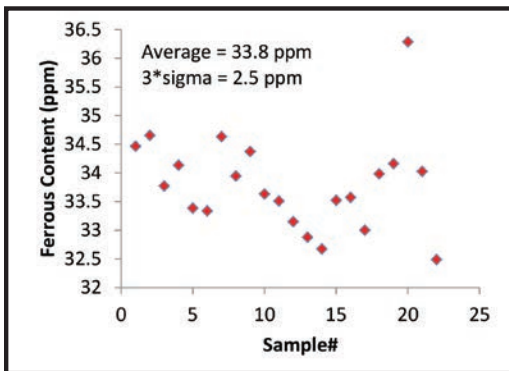


Figure 3: Example of repeatability of 20 consecutive measurements.

Case Studies

The following two case studies further demonstrate the capabilities of LNF.

Case #1: Engine Test Cell

In this example, a used oil sample is retrieved from an engine during its break-in period and shows the synergy between LNF and other techniques such as spectrometric wear metal analysis and analytical ferrography. LNF results clearly depict the typical and expected high levels of large wear particles during break-in.

The bar graph of the wear summary screen in Figure 4-3 shows the large number of particles less than 15µm in size. The number of particles greater than 20µm is shown in the cutting, severe sliding, fatigue and nonmetallic wear categories.

An LNF image map of particle silhouettes for this sample is shown in Figure 4-4. The majority of large particles are identified by LNF and quantified in the wear summary as severe sliding and fatigue particles. This fact is confirmed by conventional analytical ferrography shown in Figure 4-6. Spectrometric oil analysis of this sample also shows a high level of wear metals including aluminum, copper and silicon.

In this case, LNF clearly confirmed that the particles were formed during the engine break-in process, and are therefore considered normal wear. The close agreement between spectrometric, ferrographic and LNF data illustrates that the LNF instrument can identify an active machine wear mode or mechanism without the expense or subjectivity of a complete ferrographic analysis. However, if identification of the root cause of the problem is required or further corroboration is needed, we recommend a complete Ferrography analysis.



Figure 4-3: Wear Summary Screen

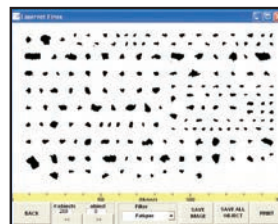


Figure 4-4: Image Map for Engine Test Cell Sample

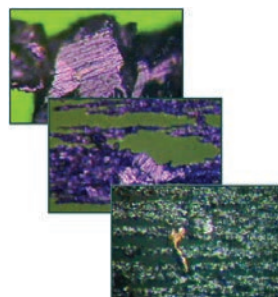


Figure 4-5: Ferrograms Showing Severe Sliding Wear and Copper Particle during Break-in

Case #2: Gearbox Accelerated Failure Test

Accelerated gearbox failure tests were conducted at Pennsylvania State University on their Mechanical Diagnostic Test Bed (MDTB) Facility under the CBM (Condition Based Monitoring) program. These tests were conducted on single-reduction 10 hp gearboxes. The gearboxes were run-in for approximately four days at maximum normal load provided by an electric generator on the output shaft. After that, a 3X over torque was applied and the system then ran to failure. The system was stopped approximately every two hours for bore site inspection and oil sampling.

In Figure 4-6, histograms of the total particle concentrations are shown for different particle size ranges. Corresponding bars in the four size ranges are from the same sample. Oil samples were drawn at successive times during the test as indicated in the figures. A similar set of data for the particles classed as fatigue, severe sliding and cutting wear are shown in Figures 4-7, 4-8, and 4-9, respectively. All particle concentrations are corrected for fluid dilution because the gearbox lubrication level was topped off with clean oil to replace each extracted sample.

The first sample was taken at the end of the run-in period, with successive samples taken during over torque operation. The sample location was changed between the 2 p.m. and 4 p.m. samples, accounting for the change in total particles counted at those two sample times. Near the end of the test, several teeth on the output gear broke before the 5 a.m. sample.

In Figure 4-6, the total particle concentration in the 5-15 μ m size range shows a general decrease during the run, which was due to gradual removal of debris generated during the run-in period as samples were drawn and replaced with clean fluid. In Figure 4-7, however, an increasing concentration of fatigue particles are seen in several of the size ranges after the 3X over torque was applied. This behavior is apparent well in advance of the ultimate failure and is probably related to the excess wear conditions that lead to failure. Similar increases in the concentration of severe sliding and cutting wear particles were not seen in any of the size ranges (Figures 4-8 and 4-9). An increase of fatigue particles is expected in an over torque situation where excessive force is concentrated along the gear pitch line where rolling action occurs.

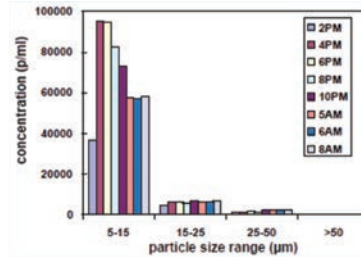


Figure 4-6:
Gearbox
Total Particle -
Concentration
Distributions

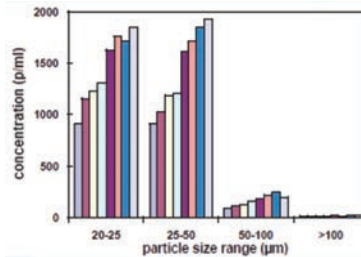


Figure 4-7:
Gearbox
Fatigue - Particle
Concentration
Distributions

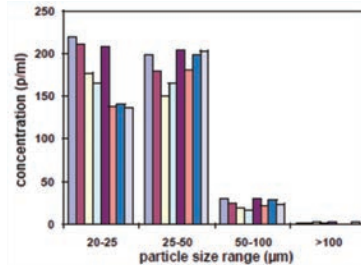


Figure 4-8:
Gearbox
Severe Sliding
Wear - Particle
Concentration
Dilutions

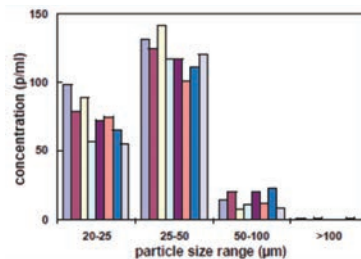


Figure 4-9:
Gearbox
Severe Cutting
Wear - Particle
Concentration
Dilutions

Conclusion

LNF is a unique analytical instrument and method that combines automatic particle shape classification and particle counting, two essential functions of used oil particle analysis.

As the case studies illustrate, by combining these two features, early signs of potential problems can be detected through increases in overall particle concentrations, and at the same time, the possible root cause of the problem can be diagnosed from shape classifications. Similar to complete analytical Ferrography, (without reflected light capability) LNF offers a unique insight into active machine wear, without the qualitative subjectivity and potential cost of comprehensive ferrographic analysis.

It provides the ideal screening tool for analytical Ferrography, allowing sample data to be trended on a sample-to-sample basis. The LaserNet Fines® Q200 series instrument provides a dynamic solution to your detailed oil analysis needs while realizing substantial cost-benefits compared to Optical Particle Counters and traditional ferrography techniques.

References

1. Thomas Barraclough, Malte Lukas and Daniel P. Anderson, "Comparison of Wear and Contaminant Particle Analysis Techniques in an Engine Test Cell Run to Failure" Pages 1-11
2. Malte Lukas, Daniel P. Anderson, Thomas Sebok and David Filicky, "LaserNet Fines® – A New Tool for the Oil Analysis Toolbox" a reprint from "Practicing Oil Analysis" Magazine
3. C. Holloway, T. Sebok, D. Filicky, J Reintjes, J.E. Tucker, P.L. Howard, "Beyond Particle Counting – LaserNet Fines® Optical Wear Debris Analyzer," Pages 1-14
4. Mark Smith, Analysts, Inc., "Oil Analysis vs. Microscopic Debris Analysis – When and Why to Choose," Pages 1-9

Chapter 5: Infrared Spectroscopy

It is well-known that infrared is an extremely versatile technology for oil analysis. IR can provide information on a range of oil characteristics, e.g. contamination, breakdown, additive packages, fluid identity, etc. In all of these cases the response of the oil to specific regions in the infrared spectrum is examined and weighted, each being unique to the characteristic being analyzed.

Infrared spectroscopy of lubricants relies on a very simple method. You observe how much Infrared radiation the lubricant absorbs as a function of the frequency of that radiation. Figure 6-1 shows such spectra for typical lubricants. That is all we need from the infrared spectroscopy itself – we just need to make sure that an accurate infrared spectrum is acquired. As you can see in Figure 6-1, different lubricant types and in general different lubricants can have very different spectra. It is these differences we use to turn these spectra into usable information (lubricant properties listed in Table 6-1).

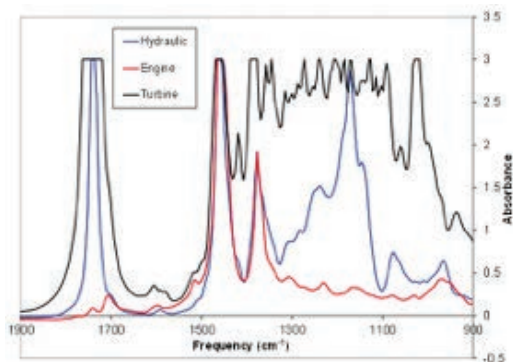


Figure 1: Typical lubricant infrared spectra. The interpretation of these spectra allows for monitoring the relevant properties listed in Table 6-1.

Fourier-Transform Infrared Spectroscopy (FTIR) is a general purpose tool for generating an infrared spectrum that has become widespread across multiple industries, including for oil analysis. In fact, the terms IR and FTIR have become interchangeable for many people. FTIR in general provides a highly accurate infrared spectrum of the sample to be analyzed. However, the FTIR can only generate the IR spectrum. It cannot analyze the spectrum. The value of IR oil analysis come from properly interpreting this spectrum. The same is true in any industry. Spectro Scientific's focus is on the analysis of the infrared spectrum to deliver quantitative results for oil analysis. This extends down to the hardware utilized for gathering the infrared spectrum: Spectro Scientific uses a grating-based spectrometer in its FluidScan product line, in addition to filter-based spectroscopy for other targeted applications. These approaches specifically target oil analysis, while other approaches are typically used for more general analysis. The advantages to choosing this approach include enabling rugged, no moving parts, low-power, and miniaturized hardware. Hardware designed specifically for oil analysis must be good enough to gather the infrared spectrum needed for a particular application. Focusing just on specific use cases allows us to design hardware that has other significant advantages, such as ease of use, portability or ruggedness.

As discussed, the power of IR is in the analysis itself, which can examine specific oil analysis properties such as % water, TAN (mgKOH/g), and fluid identity (eg., "it is highly likely that your fluid is Shell Rotella T1"). This is where the Spectro Scientific approach is unmatched in the industry. We have spent years gathering a large library of both infrared spectra and associated chemical properties, currently numbering approximately 700 different oils and greases. The importance of this cannot

Property	Type	Infrared Relevant?
Particle Count and Distribution	Contamination/ Machine Wear	
Wear Metals	Machine Wear	
Glycol	Contamination	✓
Dissolved Water	Contamination	✓
Emulsified Water	Contamination	✓
Incorrect Lubricant	Contamination	✓
Alien Fluid	Contamination	✓
Fuel	Contamination	✓
Viscosity	Contamination/ Breakdown	
Nitration	Oil Breakdown	✓
Sulfation	Breakdown	✓
Oxidation	Breakdown	✓
Soot	Contamination/ Breakdown	✓
Acid Number (AN)	Breakdown	✓
Base Number (BN)	Breakdown	✓
Antiwear Additive	Depletion	✓
Antioxidant Additive	Depletion	✓

Table 1: Typical lubricant parameters that are measured and the relevance of infrared spectroscopy per property

be overstated: First, it enables users of the FluidScan devices to automatically compare their oil of interest to this library to ensure that they have the correct fluid or even to search the library for similar fluids. Second and perhaps even more importantly, each fluid in the library has been analyzed chemically in order to establish its correlation to laboratory-based techniques for oil analysis. Prominent examples are for acid (TAN/AN) and base number (TBN/BN). This ensures that the FluidScan, when used to analyze a customer's oil, reports the relevant properties for that oil, and reports them accurately. "Under the hood" are entire families of calibrations that relate (eg., TAN to the infrared spectrum for specific chemical class of oil. These calibrations, as relevant to that particular oil, are attached to each of the 700 oils and greases. For example, there are over 30 TAN calibrations in the library, but each individual oil is assigned a single TAN calibration based on its chemical class. Without this family of calibrations, generated over years of analysis, it would be impossible to provide an accurate TAN value just knowing the infrared spectrum. The same holds true for FTIR or any type of infrared analysis. IR analysis in general can provide information and analysis well beyond standardized methods. The issue has historically been that the methods are limited to a certain set of oils. Spectro Scientific's multidimensional library approach (oil IR spectrum+chemical properties) eliminates that shortcoming. Following are more specific discussions of these features.

5.1: Direct Infrared Spectroscopy Based on Grating Optics

5.1.1: FluidScan Introduction

Oil analysis is a critical predictive maintenance technology. Service or reliability professionals, whether managing a truck or locomotive fleet, a marine vessel or fixed assets in an industrial plant, rely on oil analysis information to schedule maintenance actions. The need to continuously improve uptime and reduce maintenance costs demands in situ oil analysis results for better decision-making using oil analysis data as well as other predictive maintenance technologies.

However, performing on-site oil analysis is difficult due to the following challenges:

- Lack of in-house expertise
- Insufficient oil analysis instrumentation
- Logistics of handling hazardous chemicals and waste recycling

The FluidScan device, was designed to address the above

mentioned challenges faced by reliability professionals. It is a handheld infrared oil analyzer that delivers fluid condition assessment based on ASTM International and JOAP (Joint Oil Analysis Program) standard practices. It protects machinery by determining when a lubricant needs to be changed due to excessive contamination, degradation or fluid mix ups, considered the most common causes of oil changes. FluidScan detects lubricant degradation and contamination by other fluids (water, glycol, incorrect lubricant) at the point of use by measuring key oil condition parameters in both synthetic and petroleum-based lubricants and fluids.

The FluidScan analyzes lubricants and fluids using infrared spectroscopy, a technique that has found wide acceptance as a primary test for contamination and degradation. The device performs, displays and stores the analyses with the same accuracy as laboratory instruments, but does so on-site in a handheld version. The analysis information stored on the device's database can be synchronized with the FluidScan Manager software, a powerful database analysis package which runs on a personal computer and archives and trends data and generates fluid condition reports.

The FluidScan lubricant condition monitor is applicable to any mechanical system where unexpected downtime, as a result of lubricant degradation and/or contamination, is unacceptable. It enables operators of power generation and mining equipment, marine vessels, trucks, wind turbines, military vehicles and aircraft, heavy construction equipment, or any large industrial system, to establish predictive maintenance programs based on oil condition rather than on a pre-set time or distance schedule.

The FluidScan provides immediate on-site analysis of lubricant properties, and accurately warns the user when it is time to change the lubricant due to contamination or degradation. The primary benefits of real-time, on-site analysis include:

- Extended oil change intervals
- No waiting for laboratory analysis results
- Reduced operational and maintenance costs
- Reduction of unscheduled maintenance outages
- Prevention of catastrophic failures



Figure 7-1: FluidScan Device

Patented Optics and Flip Top Cell

The FluidScan monitor is a self-contained handheld analyzer that delivers instant fluid condition assessment to the user. It eliminates sample preparation and time-consuming cleanup using a patented flip top sampling cell for easy and rapid on-site analysis as shown below.

At the core of the FluidScan is a patented, mid-infrared spectrometer with no moving parts. The spectrometer collects the infrared light transmitted through the fluid in the flip top cell into a waveguide as illustrated below.

The waveguide then carries the light to a prism-like diffraction grating that reflects the light into a high-performance array detector which registers the infrared spectrum of the fluid. The waveguide completely contains the infrared signal, minimizing any atmospheric interference and maximizing the amount of light within the spectrometer. In this way, the FluidScan maximizes optical throughput and spectral resolution in a palm-sized device. Consequently, it provides more than adequate spectral range, resolution and signal-to-noise

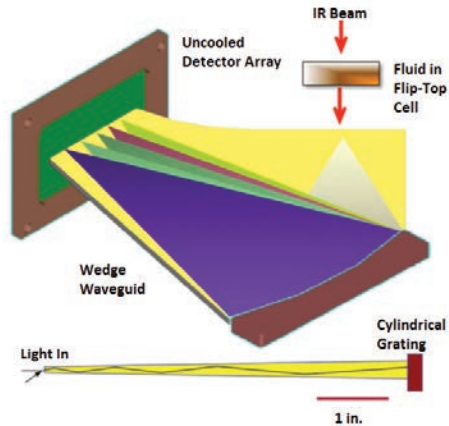
Figure 7-2: Patented flip top cell eliminates the need for solvents to clean



ratio for the rapid analysis of in-service lubricants. This unique technology has been optimized for low power consumption, enabling the production of a rugged, highly accurate miniature device that operates on Li-Ion batteries for up to eight hours.

Key infrared signatures of fluid condition, established by industry norms and ASTM condition monitoring standards, are used to obtain fluid status in real time. The user loads a sample into the flip top cell, enters sample information, and initiates an analysis using FluidScan's intuitive user interface and navigation pad. Status and supporting fluid condition parameters are then determined and displayed to the user and can be stored for trending and exporting to a central database. The information stored on the FluidScan device resides in a SQL database and can be synchronized and downloaded to a PC using the FluidScan Manager database software. This software provides data logging, trending, warning and alarm condition alerts. The FluidScan can operate without ever needing a PC, but the FluidScan Manager desktop application makes data entry and reporting easier.

Figure 7-3: Patented wedged optical design



Multivariate Calibration and Data Correlation to Laboratory Results

Most oil analysis users will compare FluidScan results to those output from a traditional off-site laboratory. Most laboratories report lubricant condition parameters using a combination of benchtop FTIR spectrometers and wet chemistry titrators.

One of the advantages of the FluidScan over laboratory FTIR is its ability to report absolute quantitative results for critical properties such as TAN and water contaminations for industrial lubricants or TBN, water, glycol, and soot for engine oils. It is only possible to obtain good absolute quantitative results through infrared spectroscopy by referencing both the correct fluid type and a calibration for that type of fluid property, as is provided with the FluidScan.

The FluidScan classifies fluids into groups called families based on their chemical makeup, usage and spectral signature. The spectrum of all fluids in each family changes in a similar way with a given amount of degradation or contamination. Family-specific algorithms are assigned that accurately quantify these amounts. These algorithms yield quantitative results for the most critical properties for the most common oil types. Multivariate calibrations are applied so that quantitative readings can be obtained even with complex contaminated samples. Our research, development and applications group has produced an extensive library of spectra from a

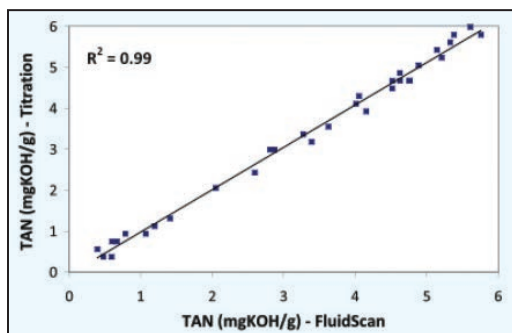
Table 7-2: FluidScan parameter settings by oil type

Oil Category	Properties measured by Fluidscan
Transmission	Water (PPM), Oxidation (Abs/0.1mm)
Hydraulic - Fire resistant (Phosphate Ester)	Water (PPM), TAN (mg KOH/g)
Hydraulic - Aerospace	
(Synthetic Hydraulic Fluid)	Water (PPM), Oxidation (Abs/0.1mm), Alien Fluid mineral based (MIL-H-2304) (%), and Alien Fluid engine oil (MIL-H-23699) (%)
Heat Transfer (Quenching Oil)	Water (PPM), Oxidation (Abs/0.1mm)
Industrial (Steam and CCGT Turbine, Hydraulic, compressor, Chiller, Gear, etc.)	Water (PPM), Oxidation (Abs/0.1mm), TAN (mg KOH/g)
Turbine Aerospace (Synthetic Gas Turbine Oil)	Water (PPM), TAN (mg KOH/g), Antioxidant (% depletion)
Engines (Engine oil for different engine types, including Gasoline, Diesel, Heavy Duty Diesel, HFO, Natural Gas, etc)	Water (PPM), Oxidation (Abs/0.1mm), TBN (mg KOH/g), Sulfation (Abs/0.1mm), Nitration (Abs/cm), Soot (%), Glycol (%), Anti Wear (%)
Ethanol in Gasoline	Ethanol (%)
FAME in Diesel	FAME (%)
Biodiesel Feedstock	Water (PPM), FFA %
Biodiesel	Water (PPM), TAN (mg KOH/g), Total Glycerin (%)

Table 7-1: Key FluidScan parameters and corresponding ASTM protocols

FluidScan Property	Reference Lab Method
AW Additives	ASTM D7412/E2412 (FTIR)
Oxidation	ASTM D7414/E2412 (FTIR)
Sulfation	ASTM D7415/E2412 (FTIR)
Nitration	ASTM D7624 (FTIR)
Glycol	ASTM E1655 and E2412
Soot	ASTM D5967 (Thermo-Gravimetric Analysis)
TBN	ASTM D4739 (Titration)
TAN	ASTM D664 (Titration)
Water	ASTM D6304 (Karl Fischer Titration)

Figure 7-4: In-Service turbine oils Total Acid Number chemical titration vs. FluidScan



large database of commonly used lubricants. Chemometric techniques are used to automatically subtract the presence of interferents in a given calibration. The TAN, TBN and water are calibrated directly to wet chemistry titration readings. Soot is calibrated to soot percentages determined by thermo-gravimetric analysis. Glycol and incorrect fluid percentages are calibrated to samples prepared with known concentrations of glycol and incompatible fluids. Table 7-1 summarizes key FluidScan properties and the ASTM protocols to which they correlate as a result of this calibration process.

To apply the correct algorithms to a particular sample, the spectrum of a new oil of the same type as the in-service oil to be tested, is measured on the FluidScan. The new oil is analyzed with spectral matching software and the best possible match is made of the unknown sample with lubricants already in the FluidScan's database. The algorithms associated with that lubricant can then be applied to samples of that oil at any stage in its service life to produce quantitative results. Figure 7-4 shows an example of data correlation between the laboratory and FluidScan for TAN of in-service turbine oils.

Alarm Limits and Reference Oil Library

In addition to reporting quantitative values for these properties, the FluidScan provides the results in an easy to understand "go", "no go" format. This is done by using absolute warning and alarm values for each property. The report uses a simple green, yellow and red system to indicate fluid within limits, near alarm state and over alarm limits (Figure 7-5).

The system is pre-set with warning and alarm limits for properties associated with each of the fluids in the database. These default alarms are based on industry best practices. All warning and alarm limits are completely customizable. Every limit can be set with

an upper and/or lower bound. They can be modified to comply with equipment manufacturer's recommendations or particular applications. The FluidScan software has a system that allows the user to define pieces of equipment as assets in the device's onboard SQL database. Each asset can be defined with its own set of property limits. Sample measurements are then saved and associated with that piece of equipment.

FluidScan comes with a built in reference oil library optimized for automotive, marine, railway, military and industrial applications. The total library includes over 450 mineral and synthetic lubricants of over 60 brands and growing. Each lubricant comes with starting values corresponding to laboratory measurements or data sheets. A key feature of FluidScan is the Validate Fluid function that matches the spectrum of any clean sample with those in the reference library. Using this function, one can easily identify an incorrect lubricant before it is added into the system.

Figure 7-5: Go/No-go results based on alarm limits in the reference library

Measure Fluid > Results	
Sample ID: deer	
Chevron Thuban GL4 SAE 140	
04 Jan 2013 10:16:56	
Oxidation	29.6 abs/mm2
TAN	5.28 mgKOH/g
Water	0 ppm
<< Export Discard Save	

Conclusion

In summary, the FluidScan's combination of patented IR technology, sampling flip top cell, unique calibration algorithms and large reference library with built-in alarm limits puts the power of FTIR, Karl Fischer Titration and TAN/TBN Titration into the hands of reliability engineers. This is accomplished without the need for lengthy measurement processes, expensive chemicals or a trained chemist to run the tests. It simplifies on-site oil analysis and enables reliability engineers to perform oil analysis as they do vibration analysis and thermal imaging. By carrying the FluidScan with them on a maintenance route, information from these three key technologies can then be easily integrated for better predictive maintenance decisions.

5.1.2: TAN-TBN Measurement Using Infrared Technique

Introduction

Total Acid Number (TAN) and Total Base Number (TBN) values are commonly used to measure degradation of machine lubricants in order to avoid machine wear, corrosion, varnish, clogged filters and other problems. Electrochemical titration has been the traditional method for measuring these values. However, this method is complex and costly, requiring relatively large amounts of sample, well-trained technicians, and disposal of hazardous reagents, among other difficulties.

This paper explores IR spectroscopy as an alternative method for measuring TAN and TBN values, using a three-step process of library building, sample classification, and multivariate regression techniques. The authors present the principles of IR spectroscopy as it relates to TAN and TBN measurements, advantages of the method, and specific examples of its application for various types of engines and lubricants.

Why Measure TAN and TBN?

It is an unavoidable fact – machine lubricants degrade over time. Thermal and mechanical stress, coupled with the inevitable contaminants, cause oil to reach a point where it can no longer perform its function as a lubricant. Degradation of a lubricant is primarily caused by the breakdown of chemical components in the oil which results in the formation of acidic by-products, formed by chemical reactions between the oil's base stock and additive components in an oxygen/nitrogen rich environment under extreme thermal stress. These acidic by-products lead to corrosion of oil wetted machine parts and the formation of varnish and sludge which can quickly clog oil filters.

It is therefore critical to monitor the level of acidity in the oil as it ages. The level of acidity is obtained by measuring either the acid in the oil (TAN) or the reserve alkalinity remaining from additives in the oil (TBN).

Traditionally, TBN is used as an indication of alkaline reserve in diesel engines and TAN is measured directly in other applications like gears, hydraulics and turbines where acid formation is somewhat less aggressive than if the acid in the oil is measured directly. TAN and TBN are reported based on the amount of potassium hydroxide or equivalent needed to neutralize one gram of the sample (mgKOH/g).

Diesel engine applications are one of the most stressful on a lubricant, as the high temperatures and thermal stresses in the engine lead to the rapid formation of acidic by-products. Thus, oils used in diesel engines need to be equipped with a high reserve alkalinity additive package that consumes the acids created before they form sludge and varnish products and corrode machine components. This is why TBN is monitored in diesel engines.

In marine diesel applications, the TBN is affected more by the type of fuel used rather than by oxidative by-products depleting the additive package. This is because the international marine shipping industry has far less strict sulfur requirements on the fuels they use, resulting in the lubricating oils requiring heavy additive packages with a high starting TBN value. In some extreme cases, the heavy sulfur fuels require the starting TBN value to be as high as 70 mgKOH/g. The reaction between water and the high levels of sulfur in the fuel creates sulfuric acid which needs to be continually monitored by TBN in this application. It is common for marine diesel engines to be checked on a weekly basis for TBN and topped up with fresh makeup oil to keep the additive package level high.

The Role of IR Spectrum Analysis

Both the basic additives in engine oils and the acid build up in other types of fluids can be seen as changes in the infrared spectrum of a sample. As seen below, TBN changes are indicated in the IR spectra as decreases in absorbance peaks related to the basic additives that are present in the engine oils, as well as changes to standard degradation peaks. The additives most typically used are calcium or magnesium sulphonates, phenates and salicylates. All of these have peaks in the 1000 and 1900 cm^{-1} region of the infrared spectrum.

The increase in TAN value for a lubricant is due to the build-up of organic acids, frequently carboxylic acids, created by the oxidative breakdown of the base oil. Oxidation products typically show absorbance peaks in the IR spectrum at 1600-1800 cm^{-1} . This can be seen in the figure below where the increasing peaks in the vicinity of 1600-1800 cm^{-1} indicate increased oxidation, nitration, and acid oxidation products. While there is a complex mixture of acids generated, there is not a single absorption peak that correlates directly with TAN, but subtle changes across the spectrum can be detected and used to establish a correlation for TAN.

Figure 9-1: Spectra for oils with TBN values between 1-16 mgKOH/g, illustrating the complex behavior of the various additive and degradative infrared peaks

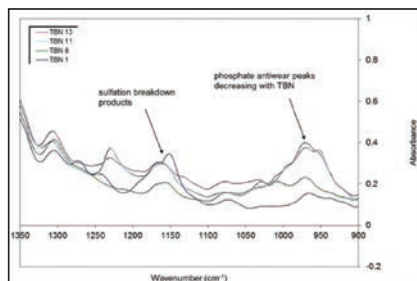
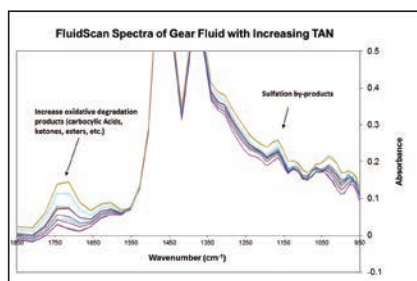


Figure 9-2: Increase of TAN in a gear oil as reflected in the infrared spectrum of the fluid. This data is taken using a Spectro FluidScan



TAN and TBN by Infrared Spectroscopy

Infrared spectroscopy, both by directly probing the lubricant “as-is” [1, 2] via relating the IR spectrum to a titrated TAN or TBN value using multivariate methods, and by measuring the lubricant’s response to a chemical reaction [3], has been used for some time to determine TAN or TBN of lubricant samples. The experimental setup involves measuring the absorbance of the lubricant through a 100 to 200-micron transmission cell using an empty cell background. The IR approach for measuring the TBN of reciprocating engine oils, due to their relatively uniform chemical composition, has been reported most commonly and is in use by some analysis laboratories as a TBN screening technique [4].

Still, questions on this subject continue to persist due to the lack of standard methods, as well as the overall chemical complexity of the lubricants themselves, which can make a “one size fits all” direct infrared method difficult. To date, only lubricant-specific calibrations for particular end user applications are, in general, widely considered quantitative.

We have approached this problem in three steps: First, we gathered hundreds of new and used lubricants of widely varying type and level of degradation into a sample library. We then recorded their infrared spectrum along with their TAN and/or TBN value using a standard ASTM titration technique (D4739 for TBN and D664 for TAN). Second, by using a standard multivariate classification technique called SIMCA (Soft Independent Method of Class Analogy), we classified each oil type based on its infrared spectrum into distinct chemical “families”. Finally, we used either the Principle Component Regression (PCR) or Partial Least Squares (PLS) multivariate regression method to relate the known TAN or TBN to the infrared spectrum, each within a given chemical family. This results in a set of family-specific TAN- or TBN-to-IR calibration curves which show quantitative correlation across a broad range of lubricants, including gear, industrial, marine diesel, turbine, and reciprocating engine fluids.

The end-result is that by carefully classifying the chemical composition of the lubricant before it is processed for TAN or TBN, one can arrive at quantitative readings using infrared spectroscopy.

Spectro FluidScan Q1000 Infrared Spectrometers

Obtaining TAN and TBN through infrared spectroscopy can be achieved, in principle, with a wide variety of approaches to obtaining the spectrum, from the now-prevalent FTIR method, to grating instruments, to emerging technologies such as tunable quantum cascade lasers. The only requirement is that quality spectra with sufficient resolution can be obtained between approximately 900-1900 cm^{-1} and 2500-4000 cm^{-1} , the areas over which the TBN and TAN calibrations can be obtained. Spectro Scientific's FluidScan, a handheld product targeted towards field applications has been designed to provide quality TAN and TBN results. The Q1000 is targeted towards end-users who need easy-to-use but accurate and reliable equipment on-site.

Table 9-1: FluidScan capabilities

Property	FluidScan Q1000
Measurement Principle	Grating
TBN Calibration range (mg KOH/g)	0-60
Repeatability, TBN relative to D4739	Better
Reproducibility, TBN relative to D4739	Comparable
TAN Calibration range (mg KOH/g)	0 to 20
Repeatability, TAN relative to D664	Comparable
Reproducibility, TBN relative to D664	Comparable
Standard Range (cm^{-1}) TAN	900 to 4000
Resolution (cm^{-1} at 1000 cm^{-1})	20
Ambient Operating Temperature (Celsius)	0-50
Weight (kg)	1.7
Power (Watts)	1.5
Operating Software	On-Device

Advantages and Disadvantages of Using Infrared Relative to Existing Titration Methods of TAN and TBN

Monitoring the level of acidity in lubricants as they age has traditionally been done through electrochemical titration of the lubricant sample. These methods require relatively large volumes of sample (ranging from 0.1 to 20 g), a well-trained technician, personal protective equipment and the use and disposal of flammable, corrosive, hazardous and toxic reagents (100 mL per sample, not including solvents required for washing and rinsing). These methods are time consuming: a TBN titration can take up to one hour. Time must also be budgeted for setup, sample preparation, function and quality checks on the instrumentation, in addition to clean-up and waste disposal. In contrast, the direct infrared approach developed by Spectro Scientific requires no sample preparation, no reagents, and no solvents. Only 0.03 mL of lubricant is required to perform

the analysis, and clean-up requires only a shop rag or towel. The measurement itself is completed in approximately one minute. Based on the fluid type, either TAN or TBN is reported in units of mgKOH/g.

Clearly, this infrared approach offers the potential for substantial cost savings through reduced labor, materials, and hazardous waste. At this point, the approach is predicated on having a fresh lubricant sample in order to classify its chemical family and thus provide quantitative TAN and TBN readings for the in-service or used lubricant. Once classified, however, samples of the new, in-service, or used lubricant may be analyzed continuously without the need to refer back to the new sample. Not all lubricants will be classified into a chemical family, as the library continues to be expanded. For these cases, established ASTM infrared methods must be used to assess oil degradation. However, at this point, >90% of the lubricants we encounter can be classified with the existing library. For an on-site operator, who knows and has their fresh lubricants of interest, the efficacy of the approach can quickly be confirmed by classifying their set of lubricants at the outset.

Performance of Infrared TAN/TBN Approach Relative to Existing Titration Methods

There are several ASTM standard titration methods that detail measuring TAN and TBN for lubricants. ASTM D664 covers the potentiometric measurement of TAN for petroleum products, titrating with a solution of potassium hydroxide. TBN can be measured following either ASTM D4739 (using hydrochloric acid) or ASTM D2896 (perchloric acid method). The latter is used primarily for new oils and the former for tracking the decrease in TBN for in-service oils. The stronger solvent/titrant mix used in test method ASTM D2896 will titrate weak bases and weakly basic degradation products in the sample that cannot have protective value. It is for this reason that our infrared TBN values are correlated to ASTM D4739.

The repeatability and reproducibility of these standard methods for used oils are well-documented. Repeatability is the variability of measurements on the same sample by the same operator using the same measurement equipment. Reproducibility is the comparison of independent tests.

ASTM D664 Reproducibility	ASTM D664 Repeatability
$R = 0.44 \times$	$r = 0.117 \times$
Where x is the average of two separate TAN readings	

For an in-service sample with a TAN of 2.0mgKOH/g, the comparison of independent tests (reproducibility) can yield results between 1.12 and 2.88mgKOH/g, with repeatability between 1.77 and 2.23 mgKOH/g.

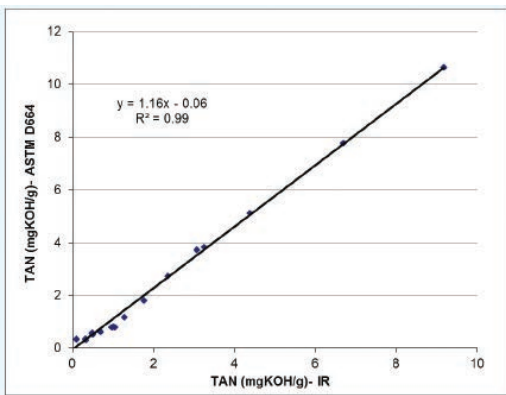
For comparison, the IR methods show typical TAN reproducibility relative to an ASTM titration measurement of 0.49 mgKOH/g at a 99% confidence interval in the normal operating range of TAN below 4 mgKOH/g. Thus for a nominal sample with 2 mgKOH/g, the results can be expected to range between 1.51 and 2.49 mgKOH/g, comparable to the ASTM method.

Repeatability is calculated at 6.8% RSD at the 99% confidence interval at the mid-range of the measurement window (0-10 mgKOH/g), which is similar to the ASTM method.

A typical calibration curve for turbine oils is shown below. The figure depicts several different brands and states of lubricant breakdown.

For an in-service sample with a TBN of 10 mg KOH/g, titration results can range from 5.5 to 14.5 mg KOH/g

Figure 9-3: Relationship between ASTM D664 and infrared TAN values



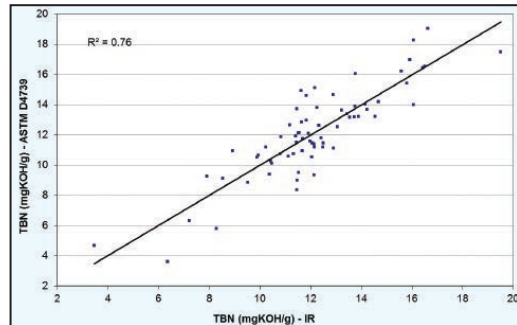
within the specification, with repeatability between 9.6 and 10.4 mg KOH/g.

For TBN, both IR methods show typical reproducibility relative to an ASTM titration of 3 mg KOH/g at a 99% confidence interval over a range of 0-16 mgKOH/g. For a sample with a nominal TBN of 10 mgKOH/g this implies one would encounter a range of results between 7 to 13 mg KOH/g, comparable to the ASTM method at this common level of TBN for new engine oils. Repeatability is 0.37% RSD at a 99% confidence interval, better than the ASTM D4739 method.

ASTM D4739 Reproducibility	ASTM D4739 Repeatability
$R = 1.54x^{0.47}$	$r = 0.22x^{0.47}$
Where x is the average of two separate TBN readings	

The figure below shows a calibration curve for reciprocating engine oils, based on a variety of different oil brands and states of degradation.

Figure 9-4: Relationship between ASTM D4739 and infrared TBN values

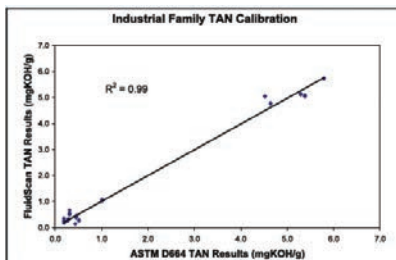


Example of TAN Correlation: Industrial Fluids

In order to illustrate the effectiveness of this direct infrared approach to providing TAN/TBN values, we investigate the case of the “Industrial” fluid family. The lubricants in this family are a collection of transmission and hydraulic fluids as well as some gear oils. They all have base oils and additives that yield fluids with high shear stability and especially good low temperature flow properties. Despite differences in base oil and additive composition for the areas that show changes with increasing TAN, the IR spectra of these fluids are similar and consistent with the SIMCA classification of these fluids into one common family. The acid build-up that raises the TAN shows up in the same fashion in these fluids. This allows a single calibration to be used to calculate a TAN value from the IR spectra of the sample. The calibration is generated from one fluid and then verified against other fluids in the class and in-service samples when available. The calibration approach starts by choosing the most “central” fluid in the SIMCA classification, i.e. a fluid that has a classification score in the middle relative to other members of the family. Once the fluid is chosen, a matrix of degraded samples is generated from that fluid. The sample matrix encompasses the range of expected TAN values, degradation and contamination that would be expected in a real world application of these types of fluids. For this type of fluid, a range of oxidative degradation is created and various amounts of water are homogenized into the samples. The TAN values for all of these samples are titrated following ASTM D664, and infrared spectra of each sample are acquired. PLS or PCR methods are then employed to create the calibration between the IR spectra and the titrated values. The results are shown below, which indicate that an excellent calibration curve relating the IR spectrum to TAN is generated.

Since the calibration must be robust across the range of samples, the calibration is tested against other fluids in the same family. A set of verification samples is generated similar to the calibration set described above. TAN values for these samples are obtained via ASTM D664 and the newly-created infrared TAN calibration. As seen in the correlation data in Figure 9-6, the TAN of a transmission fluid can be predicted well using the calibration for the Industrial family.

Figure 9-5: Industrial Family TAN Calibration using the FluidScan



Example of TAN Correlation: Heavy Gear Oils

An interesting calibration is that of a family of heavy gear oils which are particularly applicable for the wind turbine industry. These fluids have TAN values that initially decrease with service, as an acidic additive package is slowly depleted. Eventually, the buildup of acidic products outweighs the suppression of TAN due to the additive package and the TAN begins to rise. For these fluids, the manufacturer specifies both a low and high TAN limit, consistent with how the TAN evolves in the oil.

As seen in below (plotted as a difference spectrum relative to the clean oil), the absorbance in the 1400- 1600 cm^{-1} area decreases to a minimum level, consistent with the depletion of an additive package in the oil. The adjacent areas begin to show an increase in absorbance that grows stronger as the TAN starts to increase from its minimum value of approximately 2 mgKOH/g (from a clean oil value of 4 mgKOH/g), up to a value of 3 mgKOH/g in this dataset. The upper TAN limit is in the range of 5 mgKOH/g.

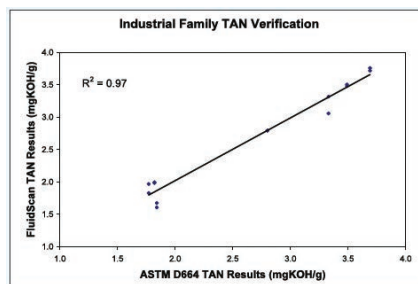


Figure 9-6: Verification of Industrial Family TAN calibration by using another fluid in the family, using the FluidScan

The figure below shows that the infrared TAN has excellent correlation to the ASTM D664-determined TAN, indicating that the infrared multivariate analysis is able to account for and properly weight the various spectral effects observed.

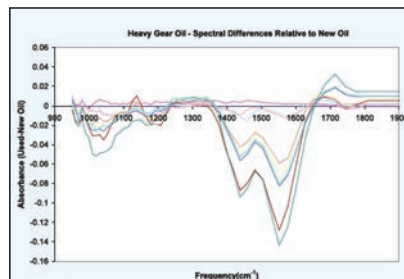


Figure 9-7: Spectral evolution of a heavy gear oil relative to the new fluid shows both increasing and decreasing absorbances at various frequencies as the oil becomes more degraded

Example of TBN Correlation: Marine Diesel Engine Oil

For the classification of marine diesel engine oils and reciprocating engine oil families, there is a strong overlap between the families for comparable TBN values. This is not surprising, as the main differentiator between the oils in both classes are the TBN-boosting additives such as detergents, soaps and dispersants. The core calibration for the marine diesel engine oil family is obtained from an analysis of a variety of in-service and used diesel engine oil samples of commercial vehicles including buses, trucks and small boats.

This calibration accurately predicts TBN changes in the oil to levels exceeding 50 mgKOH/g all the way down to full degradation. Different from the TAN examples, we were able to obtain an excellent sample matrix from the in-service and used samples without further treatment of the oil. This is always the preferred scenario, as these

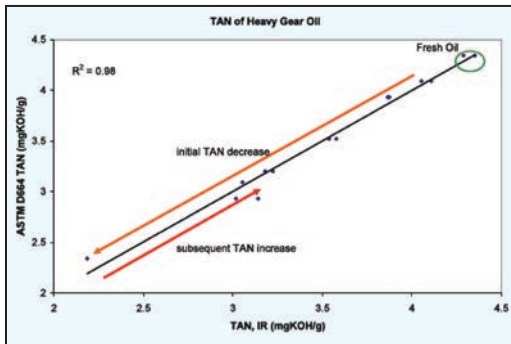


Figure 9-8: Relationship between ASTM D664 and infrared TAN values for a heavy gear oil. This oil has a complicated evolution where the fresh oil's TAN initially decreases down to a minimum level, then begins to increase towards maximum alarm value.

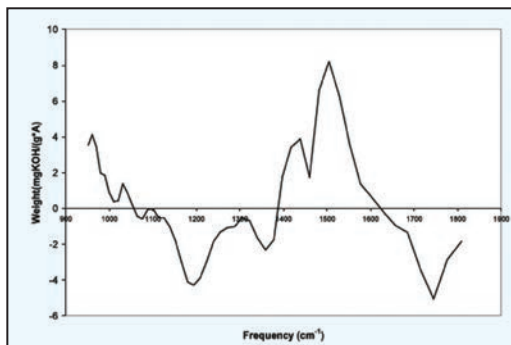


Figure 9-9: Prediction vector for the FluidScan for marine diesel engines

samples more fully represent the complex cycling the oils undergo while in service, and thus are most likely to provide accurate predictions, at least in principle. The figure below shows the TBN prediction vector for these marine diesel oils. The TBN value itself is obtained from a linear multiplication of this prediction vector with the actual infrared spectrum.

Finally, to illustrate the stability of the TBN measurement using the FluidScan over a period of time, the table below shows the TBN of five different marine diesel engines over a period from June 2010 to January 2011, as taken onboard by the FluidScan handheld device. The TBN of the oil is checked monthly and fresh oil is added to the system to maintain a steady base reserve. The on-site capability of the FluidScan is well adapted for this type of application. These results show excellent accuracy relative to off-shore laboratory titration measurements over the entire period.

Date	Sampling Point/ Equipment	Product Type	TBN mgKOH/g FluidScan	TBN mgKOH/g Titration
June 2010	No. 5 Engine	Marine Diesel	14.7	15.3
July 2010	No. 5 Engine	Marine Diesel	14.6	14.2
November 2010	No. 5 Engine	Marine Diesel	14	13.9
January 2011	No. 5 Engine	Marine Diesel	13.9	13.6
September 2010	No. 4 Engine	Marine Diesel	14.5	15.6
January 2011	No. 4 Engine	Marine Diesel	13.6	13
June 2010	No. 4 Engine	Marine Diesel	14.4	14.5
October 2010	No. 4 Engine	Marine Diesel	14.3	14.3
September 2010	No. 4 Engine	Marine Diesel	14.5	15.8
December 2010	No. 4 Engine	Marine Diesel	13.4	12.8
September 2010	No. 4 Engine	Marine Diesel	13.7	14.9

Table 9-2: Correlation between on-site FluidScan and off-shore laboratory Titration methods over a period of time between June 2010 and January 2011

Conclusion

The results presented in this paper show that it is possible, using the infrared spectrum of a lubricating fluid sample and software available on the FluidScan, to obtain reliable TAN or TBN readings for a wide range of lubricating fluids. This is achieved through a three-step process of library building, sample classification, and multivariate regression techniques, all of which are built into the FluidScan's software. The expanding library and promising results relative to traditional titration methods, indicate that this approach has wide applicability to both off-site laboratory and on-site oil analysis programs.

References

- [1] Jay R. Powell, "Molecular Condition Monitoring in the Commercial World: Objectives and Applications of FT-IR Analysis", Proceedings of the JOAP International Condition Monitoring Conference, p. 186-193, (1998).
- [2] Dave Wooton, Robert Thomas, Stuart Barry, and Samuel White, "Using Infrared Spectroscopy in Used Engine Oils – Estimating Base Number", Practicing Oil Analysis, p. 30-35 (2005).
- [3] F.R. van de Voort, D. Pinchuk, M. Davies, and A. Taghizadeh, "FTIR Acid and Base Number Analyses: Their Potential to Replace ASTM Methods", Proceedings of the JOAP International Monitoring Conference, (2002).
- [4] See, for example, Neil Robinson, "Monitoring oil degradation with infrared spectroscopy", WearCheck Africa Technical Bulletin, Issue 18 (2000).

5.1.3: Water Measurement Using the Infrared Technique

5.1.3.1: Dissolved Water Measurement

Water in oil can manifest itself in many forms – from dissolved to emulsified, to free water with oil. How water will be present in a given oil depends on the nature of the oil formulation as well as sample preparation conditions. The FluidScan mid-infrared spectrometer from Spectro Scientific is designed to monitor the presence of water in all the above scenarios. The device uses the fundamental chemistry that describes the presence of water in oil, providing a highly sensitive and quantitative measure of dissolved water in oil. By monitoring the presence of scattering in the oil from emulsification or the presence of free water, FluidScan alerts the user when these situations exist at critical levels. In comparing results from FluidScan to Karl Fischer laboratory titration methods, operators should take care to ensure the water status is homogenous. Quantitative comparisons can be made when the water is predominantly dissolved in the oil. In the following example, we discuss details of how to measure water with FluidScan.

The task of detecting water contamination in oil using a mid-infrared spectrometer such as FluidScan is a natural fit. When water interacts with oil, signature chemical bonds are created between the water and the oil molecules, which have strong mid-infrared resonances. This means the bonded water-oil molecules preferentially absorb mid-infrared energy at specific

vibrational frequencies. The natural frequencies of the bonds' vibrations cause this absorption.

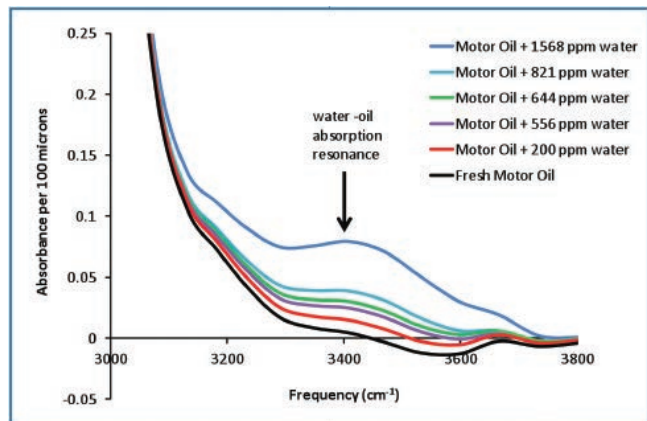
Think of the vibration of a heavy parcel attached to a spring (or multiple springs) inside a shipping container. Such vibrations will occur most prominently at the natural resonant frequency of the spring and parcel combination. The same principle applies with chemical bonds; the incident infrared light supplies the energy instead of a jolt to the shipping container as it is being loaded. For FluidScan, we are interested in a particularly sensitive oxygen-hydrogen bond resonance (O-H stretch) that occurs between the "dissolved" water and oil. Depending on the oil itself, this resonance occurs between 3200 and 3800 cm^{-1} , or 2.63 and

3.125 microns in wavelength. In most mineral-based oils this resonance will be in the vicinity of 3400 cm^{-1} . Figure 8-1 shows an example in typical, multi-weight motor oil.

As shown below, as more dissolved water is present in the oil, the absorption resonance continues to increase.

Since this process is repeatable and driven by the fundamental chemistry of the chemical bond, precise correlations can be made between the absorption resonance and the amount of water present in the oil. This is illustrated in the graph below, which shows excellent correlation between FluidScan-determined values for the water compared to Karl Fischer standard laboratory titration values.

Figure 8-1: An example of dissolved water absorption in motor oil: FluidScan spectra depicting a series of motor oil samples with increasing amounts of water. The main absorption resonance is centered at 3400 cm^{-1} .

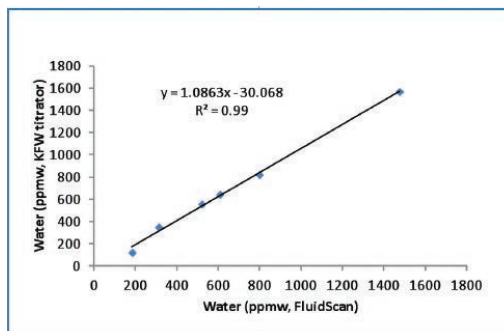


FluidScan analyzes a sample and produces a ppm reading in the following manner:

- FluidScan measures the spectrum of the oil and embedded analysis algorithms quantify that the height of the water peak is based on that spectrum.
- That height is then turned into a ppm using an algorithm stored inside the device. No matter what type of oil (or biodiesel for that matter), this signature peak is present when water bonds with the oil. This enables a straightforward and reliable way for FluidScan to probe water content in oil.
- By using an algorithm to determine the true height of this peak (similar to how your eye can), other situations in oil (such as the presence of soot) with activity in this region can be eliminated from the true water determination.
- In cases where infrared peaks occur in this same mid-infrared region, such as when a significant amount of antifreeze contamination is present in the oil, FluidScan identifies and flags other peaks associated with those chemical contaminants apart from the water contamination.

When present, however, water will not always be able to fully bond with the oil and produce an infrared absorption resonance associated with such dissolved water. In fact, this is our most common experience: “They mix like water and oil.” This means they do not mix very well at all. Fortunately, in machinery with its elevated fluid agitation, temperature and pressure conditions, water will often be able to dissolve or bond up to the saturation level of the oil. Saturation means the available sites on the oil molecules that form a water-oil chemical bond have been used up. Saturation levels can vary between 500 ppm in a hydraulic to 3000+ ppm in engine oil. Once you reach saturation, the water in the oil tends

Figure 8-2: Comparison of FluidScan water readings from the analysis of the spectra in Figure 8-1 to ASTM D6304 Karl Fischer water titration



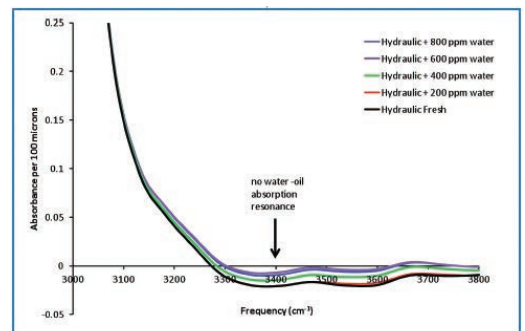
to emulsify and form small globules in the oil. In cases of extreme water contamination, free water, which can settle anywhere in the machinery, will be present. Water can also be driven to these states due to the design of the oil itself, where additives to the oil may act to prevent oil-water bonds from forming except under extreme conditions or as the oil ages.

In cases where emulsified and free water are present in the oil, these contaminants tend to scatter the infrared light passed through the oil by FluidScan. The graph below shows the example spectrum of emulsified and free water present in a water resistant, synthetic hydraulic oil.

The signature effects of scattering, rather than the absorption of infrared light by the oil-water mixture, is a broad but small elevation of the absorbance spectrum of the oil across the infrared spectrum. The infrared light scatters off the water globules in all directions. Rather than transmitting straight through the oil, infrared light is lost from the system and is recorded as a drop in transmission through the oil and it is recorded as an increase in absorbance. This situation is unstable since the water itself is inhomogeneous within the oil. For example, the emulsified water, when pulled from the machinery, may settle to the bottom of the container as free water, if it is left to sit too long. This will completely change the amount of scattering in the oil, making the quantitative determination of emulsified or free water difficult. Indeed ASTM standard Karl Fischer methods quote a wide lab-to-lab reproducibility of 700 ppmw for a 500 ppmw sample (ASTM D6304 Procedure C), even though such apparatus are more sensitive than that, partially due to this lack of stability of water in oil.

In these cases, FluidScan’s embedded algorithms search for this increase in the absorbance spectrum and warn

Figure 8-3: Infrared signature of emulsified and free water in oil: The presence of such contaminants is marked by elevated absorption across the infrared with no peak, a signature of the scattering of the infrared light by the water.



the user to the presence of the water if it is above pre-defined thresholds. An alarm of “Significant Free Water Detected” will be present on the Results screen to alert the user that this situation is occurring. It is relatively straightforward to distinguish this type of activity from true absorbance or other types of scattering (such as soot again), because they tend to affect the infrared spectrum gathered by FluidScan in a much different manner. In these situations, FluidScan will provide an

indication rather than a quantitative reading of the total amount of water, and therefore quantitative comparison to laboratory Karl Fischer titrations techniques cannot be made. A quantitative value of dissolved water in ppm, however, will still be provided by the device. Such a dissolved water value alerts the user when the oil, even if not designed to take on water, is dissolving water and reaching its saturation. However, it should not be confused with a total water ppm reading.

Conclusion

In summary, the FluidScan mid-infrared spectrometer is an excellent mechanism to detect the various manifestations of water contamination in oil.

- Dissolved water provides an unambiguous, repeatable and large absorption resonance, which FluidScan can readily quantify and track for virtually any oil type.
- The presence of emulsified and free water is also identified by FluidScan via their characteristic infrared scattering, and alerts the user when they reach a critical level. Depending on the makeup of the oil itself, either dissolved or emulsified/free water may be more prevalent.
- Many oils (such as most motor oils) are designed to dissolve as much water as possible in order to remove it from the machinery. In these cases, dissolved water peaks are readily observed and their quantification, up to the saturation point, is highly accurate.
- Other oils, including hydraulics that are apt to encounter significant water well beyond the saturation limit of any oil type, are designed for their demulsibility characteristics (i.e., their ability to reject bonding with water) so the water may be removed from the system by other means. In these cases, FluidScan alerts the user to extreme contamination conditions.

5.1.3.2: Free Water and Total Water Measurement

Synopsis

Water calibration using the FluidScan analyzer provides accurate determination of total water contamination in an oil sample of 1000 and above for all turbine oils and from 1 ppm and above for a growing number of turbine oils. The analysis provides an alternate option to the traditional Karl Fisher (KF) coulometric titration (ASTM D6304).

Introduction

Turbine oils typically are formulated to have high thermal stability, oxidation resistance, and excellent water separation. Lubricants available specifically for gas

turbines or steam turbines are designed with specific additive formulations, but there are also many oils that can work with all different types of turbines. Gas turbines have the tendency to build up sludge and varnish whereas steam turbines may experience oxidation, foaming, and sludge. However, a concern of all turbine systems is water contamination. Regular and reliable water measurement in turbine oil is an important part of successful turbine operation. Severe water contamination can cause changes in the oil's viscosity, accelerated oxidation, additive depletion, and decreased bearing life. Turbine manufacturers typically recommend a warning alarm limit of 500 – 1000 ppm.

The most widely accepted method for detecting water in oil is by Karl Fischer (KF) coulometric titration (ASTM D6304). This titration method is somewhat cumbersome, as it

requires hazardous reagents, careful sample preparation, expensive equipment, and at least several minutes per analysis. However, Karl Fischer analysis for water can yield highly accurate and repeatable results when executed by a skilled operator and is the comparative method for other analytical techniques for water determination. Also, the water does not have to be fully dissolved in the oil.

The FluidScan portable analyzer can detect the light scattering of water droplets present in oil by a lift in the baseline of the infrared absorbance spectrum. Figure 1 shows several FluidScan spectra of used turbine oil samples with high levels of water contamination.

The degree of light scattering caused by a water-in-oil mixture indeed depends on the concentration of water present, but it also is strongly influenced by how the water is physically dispersed in the oil: the number and size of discrete water droplets present in the oil (Figure 2).

For this reason, it is important to have representative, homogeneous sampling. A portable instrument such as the FluidScan can be used at the sampling site for immediate results where the oil and water will be homogeneous

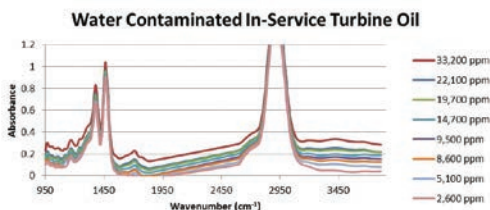


Figure 1: FluidScan spectra of used turbine oil heavily contaminated with water used to monitor a vacuum dehydration process at a power generation plant.



due to the turbulent motion inside the instrument. If the samples are left to settle, perhaps during transit to a designated oil analysis site or laboratory, the water will eventually separate (Figure 3). After the water has completely separated from the oil, it is difficult to get accurate measurement of the water content.

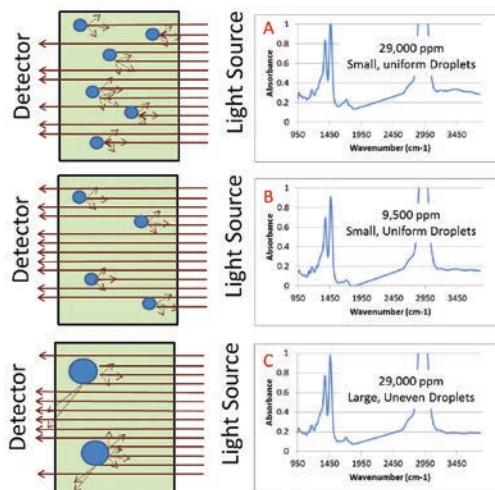


Figure 2: Graphical representation of light scattering in used turbine oil due to varied water droplets. Spectrum A is a used turbine oil with 29,000 ppm water contamination immediately analyzed after homogenization. Spectrum B is a used turbine oil with 9,500 ppm water contamination immediately analyzed after homogenization. Spectrum C is the same sample as in A (29,000 ppm) but has been allowed to sit for 45 minutes after homogenization. The change in concentration and water droplet size is apparent in the degree of baseline lift.

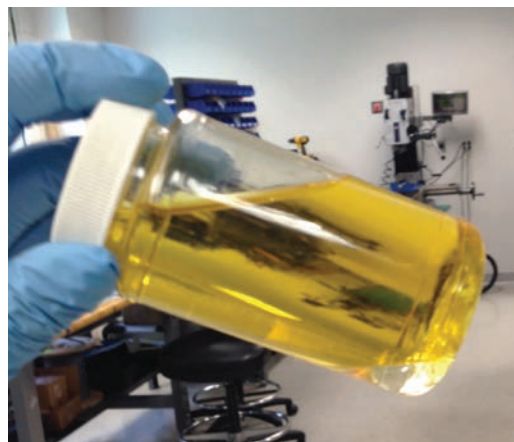


Figure 3: Sample of used Chevron GST 32, as received after shipment from a power generation plant.

Method

A new water calibration which measures light scattering due to the presence of water droplets is available on the FluidScan for the Industrial Library. The method was developed with water-contaminated samples of several popular brands of turbine and gear/bearing oils for a robust universal calibration of industrial fluids ranging from 1,000 ppm up to 65,000 ppm water. An important component of the method is the use of a homogenizer. The samples were homogenized with a CAT 120X homogenizer and allowed to sit at room temperature for 2 minutes (no more than 30 minutes) prior to measurement on the FluidScan (Figure 4).



Figure 4: Homogenizing a sample of water-contaminated oil.

Results

Sixteen samples between the range of 500 ppm and 10,000 ppm water contamination were used to test the Total Water FluidScan measurement against Karl Fischer D6304. Each sample was prepared by homogenizing them for 30 seconds on high prior to analysis. They were measured simultaneously on three FluidScans and by Karl Fischer to minimize the effects of sampling errors. The results are shown in Figure 5.

To demonstrate the importance of the homogenizer in the determination of industrial fluids which are designed for excellent water separability, a test set comprised on 13 in-service Chevron GST 32 oil samples from a power generation plant were analyzed with and without proper homogenization.

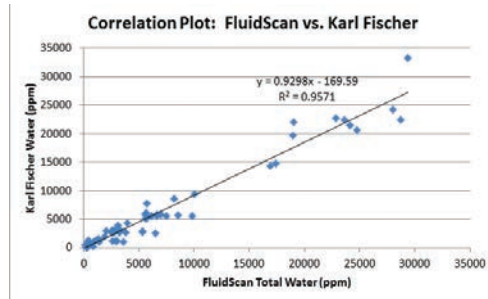


Figure 5: Comparison of the new total water measurement on the FluidScan to ASTM D6304 Karl Fischer titration method.

Group A: Samples were homogenized for 30 seconds on high (Figure 6). Before analysis, the sample bottles were gently inverted 20 times to mix.

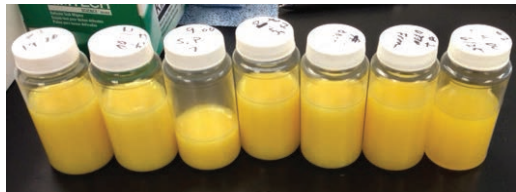


Figure 6: Samples which after being homogenized for 30 seconds on high.

Group B: Samples were shaken vigorously by hand for 30 seconds (Figure 7) and then left to sit for several minutes to allow air bubbles to dissipate. Before analysis, the sample bottles were gently inverted 20 times to mix.



Figure 7: Sample which was shaken vigorously by hand for 30 seconds. To the eye, the opacity looks similar to the homogenized samples even though the water is not uniformly dispersed in the sample.

A plastic disposable pipette was filled from the middle of the bottle, and the same aliquot was used to dispense fluid into KF vials and onto the FluidScan flip-top cell. The results are shown in Figure 8.

Clearly, the sample preparation method has a large effect on the results. All samples prepared only with vigorous hand-shaking (Method B) had unacceptably large error, and in fact, never measured higher than 6,000 ppm water on the FluidScan. Even though the hand-shaken sample appeared opaque similarly to the homogenized samples, a hand-shaken mixture of oil with water is not truly homogenous. For at site analysis, a fresh oil sample measured immediately at the sampling site should be homogeneous with uniform water droplet size from the turbulence and shearing inside the machine.

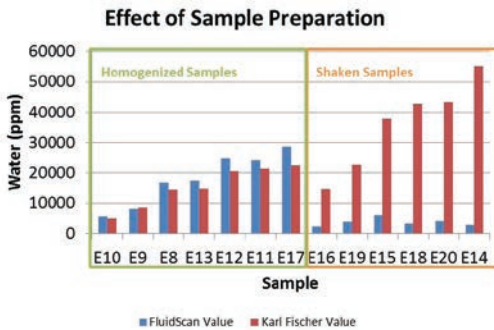


Figure 8: The samples prepared with a homogenizer showed great agreement between the calculated water concentration on the FluidScan and Karl Fischer result. The samples that were shaken by hand were not accurate.

The new, improved water measurement for turbine oils is now available on the FluidScan for all turbine oils from 1,000 ppm and above. If the total water calibration <1,000 ppm is not available for a fluid the total water detected is <1,000 ppm, the FluidScan will report the traditional E2412 dissolved water result and alert the user with a message “Free Water may be present <1000 ppm” (Figure 9). This is a benefit over the old water calibration for FluidScan because the old method just reported the dissolved water peak and left the user to wonder whether there was free water present in the oil.



Figure 9: The warning message to the user that free water may be present in a sample up to 1000 ppm.

Conclusion

The new FluidScan method for analysis of water contamination in turbine oils is a robust, reliable method capable of providing immediate alert of severe water contamination. The largest contributor to the variation is the sampling. Hand-shaking is not sufficient for obtaining a homogeneous sample and reliable results for water measurement on the FluidScan. Immediate analysis at-site or the preparation of samples prior to analysis with a commercially available homogenizer is recommended for the best results. With best practice sampling technique, results correlating within 20% to Karl Fischer can be achieved. The new FluidScan water calibration provides accurate determination of the total water contamination in an oil sample of 1,000 and above for all turbine oils and from 1 ppm and above for a growing number of turbine oils. A benefit over the previous water calibration based on E2412 is that the FluidScan will alert the user when the total water is less than 1,000 ppm if a more accurate determination cannot be provided.

5.2: Filter Based Infrared Techniques

For a large number of oil analysis applications, it is best practice to probe specific properties. For example, many laboratories use infrared analysis to screen for soot in engine oil samples as an incoming step to provide a fundamental read of the severity of contamination and potential breakdown in the oil. If the soot level is higher than a specific value, the sample may be rerouted. Infrared analysis using filters, also known as filtometry, is often an excellent, highly accurate solution in such cases. As shown in Figure 1, the analysis uses one or more filter/detector combinations, each of which detects energy at a certain bandpass in the infrared, to analyze the infrared radiation emerging from the sample. The advantages are clear and these techniques have been utilized for decades. The hardware is simple and durable, and can offer a low-cost alternative when only a limited number of properties need to be monitored.

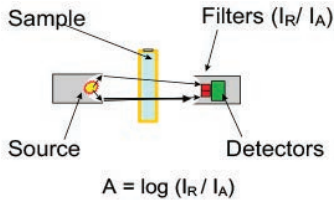


Figure 1: Schematic illustration of infrared filtometry: An infrared lamp directly illuminates the sample. The passed infrared energy is detected by a one or a number of detector elements, with a bandpass filter positioned in front of each detector.

A less-discussed advantage is the highly accurate nature of filtometry. In general filtometry is limited by the bandpass or width of the infrared filter set used. A typical filter bandpass may be 1.5% of the center wavelength. For example, at 10 micron wavelength (1000 cm^{-1}) the resolution of such a typical filter would be 15 cm^{-1} . This is in contrast to an FTIR spectrometer, which is typically utilized at 4 cm^{-1} resolution. At first glance this would seem to be a disadvantage for filtometry; however often the reverse is true. Why? For oil analysis and liquids in general (as opposed to gases) the infrared features of interest tend to absorb? over a relatively broad wavelength band. For example the infrared spectrum of motor oil, shown in Figure 2, exhibits features (peaks in the absorbance) across the infrared. However, even at significantly reduced resolutions down to 32 cm^{-1} , all of these features are still sharply defined. This gives us a hint as to why reduced resolution (reduced to a point, for example at 128 cm^{-1} resolution, most features are lost) may have advantages. So, in general, if we have enough resolution to maintain a clear read of these features, we have the advantage that the filter is gathering infrared energy across a broader spectral range, i.e. is gathering more infrared radiation. If we use the same detector, and gather more energy at each band, we are increasing our signal-to-noise ratio (SNR). This has been shown to be true for FTIRs and in general any type of spectrometer. The SNR is directly linked to the

accuracy of the measurement of the property of interest, (eg., ZDDP additive package depletion). So for optimal SNR of the obtained spectrum, we in fact WANT as low a resolution as possible. Our limitation then becomes that we have to trade-off resolution (to be able to actually see our feature) for SNR. What then is the optimal trade-off for oil analysis? How low a resolution can we tolerate before our measurement is degraded?

Mathematically, the relationship between the width of a spectral feature of interest and the resolution of the measurement is called the Resolution Parameter ρ :

Where $\Delta\nu$ = spectral resolution

FWHM = width of spectral feature of interest

Research has shown that a resolution parameter in the range of 0.5 is the most the resolution can be decreased before the measurement is degraded. For each feature of interest, a filter that gives a $\rho=0.5$ is the optimal filter setting to maximize the SNR while still providing the highest accuracy possible for a given oil property. Again, this is true no matter what type of instrument, be it an FTIR, filtometer, or grating device. Since we have many

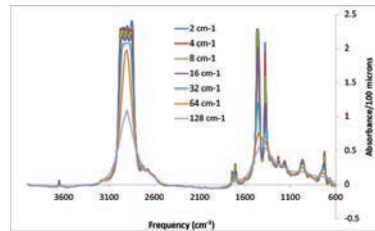


Figure 2: Infrared spectrum of motor oil as a function of spectral resolution. It can be seen that, down to approximately 32 cm^{-1} resolution, all of the essential features of the spectrum are maintained.

features of interest, we have many different peaks to examine. In filtometry we can even tailor $\Delta\nu$ for the FWHM of each feature to optimize accuracy. This is why filtometers can provide more accurate readings than FTIRs for some properties of interest even though this may seem counter-intuitive. For the determination

$$\rho \equiv \left(\frac{\Delta\nu}{FWHM} \right)$$

of ZDDP additive package depletion, for example, the feature of interest is in the vicinity of 965 cm^{-1} . The optimal filter bandwidth for this peak is 45 cm^{-1} . At a resolution parameter of 0.5, this would mean we would want to measure this additive depletion at a resolution of 22.5 cm^{-1} for optimal accuracy. In contrast to the 4 cm^{-1} resolution of FTIRs, a filtometer with a nominal resolution of 15 cm^{-1} at this frequency may provide for a more accurate reading.

We should note that this is only one example, but illustrates that filterometer systems, when appropriate for the use-case, need not be considered to be an analytical compromise.

A prominent example of filterometry in wide use by both field systems and laboratories is the InfraCal soot meter from Spectro Scientific shown in Figure 3. As indicated, soot is an essential indicator for diesel engines. This simple device monitors a single infrared region (2.9 micron wavelength with a spectral resolution of 3.8% or $\Delta\nu = 95 \text{ cm}^{-1}$) in order to determine soot content. In this case, monitoring just the one region is more than sufficient to enable accurate determination of soot in engine oils. Since soot scatters light, its feature is spread across the entire infrared spectrum, indicating that the optimal probe of the property may have a very wide bandwidth. The accuracy of this method is reflected in its associated standard, ASTM D7686. Another advantage of dedicated filterometer systems is that they can be tailored to provide performance specific to that monitored property. In the case of the Spectro Scientific InfraCal soot meter, soot values of 0-12% wt are immediately analyzed, which is appropriate for heavy-duty diesel engines. This contrasts with the standard FTIR method ASTM D7844 where only levels between 0-3% wt can be measured before you must dilute the sample with mineral spirits in order to get up to levels of 10.8% wt. Here, since the InfraCal is specifically designed for the measurement of soot, it utilizes a cubic zirconia-based attenuated total reflectance (ATR) crystal surface for its measurement. Using an ATR allows for a highly absorbing material, such as soot, to be analyzed since by the nature of the technique the infrared radiation only probes a few to a few tens of microns into the sample. Further, the crystal surface is simple to wipe clean with a moist cloth. In contrast, a standard FTIR setup limits soot levels since it probes 100 microns of material via a transmission flow cell. At 3% soot, the infrared spectrum becomes essentially 100% absorbing in the 2000 cm^{-1} range with this FTIR setup, and so accurate measurement of neat samples at 4% or higher is not possible. Further, sooty samples are hard to clean from such FTIR flow cells. This example illustrates the power and utility of dedicated filterometry.

The Spectro Scientific MicroLab also utilizes filterometry to provide a reading of seven (7) core infrared oil analysis properties, per ASTM D7417. In this case, the filterometer has many more filters (15), but the measurement principle is the same. The system is shown schematically in Figure 4. A filter wheel rotates a series of at least 15 bandpass filters into position between the IR source, sample flow cell and sample detector. The amount of infrared radiation absorbed is recorded when each filter is in place. Seven (7) oil properties are calculated from these absorbance levels. Such a filter-wheel approach has proven time and again to be a rugged, reliable method of probing for a set range of properties. In this case, the properties, listed in Table 1, are focused on monitoring of diesel-based systems as reflected in the practice ASTM E2412. The spectral resolution $\Delta\nu$ of each filter is chosen such that an accurate reading of the property of interest may be obtained. Multiband techniques, which utilize the readings from multiple filters, are used to provide an accurate measure of TBN in diesel

systems at levels below 20 mgKOH/g. In this way, a relatively simple filterometer is used to provide a range of accurate readings for evaluating diesel and related applications.



Figure 3: InfraCal soot meter from Spectro Scientific. The sample is simply squirted onto the top glass plate and a determination of soot appears in seconds on the display screen.

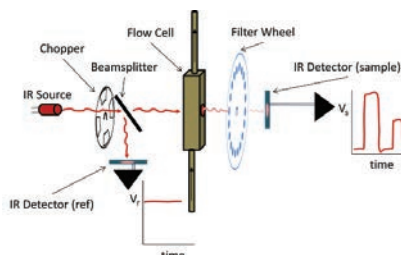


Figure 4: Schematic of MicroLab infrared filterometer. A Filter Wheel rotates a series of bandpass filters into position between the IR Source, sample Flow Cell and sample Detector. The amount of infrared radiation absorbed is recorded when each filter is in place. Several oil properties are calculated from these absorbance levels. The Chopper acts to modulate the infrared radiation to increase detector SNR. The infrared radiation emerging from the Chopper is split in two directions by a Beamsplitter. One half is directed at the Flow Cell to analyze the oil. The other half is directed to a reference Detector, which monitors the power level of the IR source. This signal is used to normalize the sample Detector signal.

Property	Units
Water	%wt
Glycol	%wt
Soot	%wt
Fuel Dilution	%wt
Oxidation	Abs/0.1 mm
Nitration	Abs/0.1 mm
TBN	mgKOH/g

Table 1: Oil Properties reported via the IR filterometer embedded in the MicroLab.

Chapter 6: Viscosity

The most important physical property of lubricating oil is viscosity. Viscosity determines the load carrying ability of the oil as well as how easily it circulates. The correct balance between high viscosity for load carrying and low viscosity for ease of circulation must be considered for any lubricant and its application. Oil provides benefits in addition to lubrication, and it is vital that it be able to flow under all conditions. When in use, contaminants such as water, fuel entering the oil, oxidation, and soot all affect the viscosity. Therefore viscosity measurement is one of the more important tests for oil in a mechanical system.

For machine condition monitoring, kinematic viscosity, defined as the resistance to flow under gravity, is the established method.

The viscosity of oil is impacted by:

- **Temperature variations** – The Viscosity Index (VI) of a lubricating fluid refers to how much the viscosity of the oil changes with temperature. A high VI indicates the oil undergoes little viscosity change due to temperature fluctuations, while a low VI indicates a relatively large viscosity change. Oil with a viscosity that does not change much between 40°C and 100°C will have a higher VI than an oil with a greater change in viscosity. The Viscosity Index Test (ASTM D 2270) is based on the Kinematic viscosity of the oil at 40°C (104°F) and 100°C (212°F). Viscosity index numbers above 95 are considered high. Oils with a high VI provide more protection to critical components over a wide range of temperatures.
- **Additives** – Additives can be part of the formulations of oils. For example, multigrade mineral-based engine oils (except naturally high VI base oils) are

formulated with a springy additive that is compact at low temperatures and expands at high temperatures in response to increasing fluid solvency.

- **Soot** – Commonly encountered in diesel engines, soot is a particle that results in a colloidal suspension in the oil. The oil's dispersant additive, designed to keep soot particles from agglomerating and growing, facilitates the formation of a colloidal suspension.
- **Water contamination** – Oil and free water don't mix, not chemically anyway. But under certain circumstances, they will combine to form an emulsion which looks like coffee with cream, and this will actually increase the kinematic viscosity of oil.
- **Thermal and oxidative degradation by-products** – These by-products are insoluble but are carried by the oil in a stable suspension.

Kinematic viscosity ν , describes a substance's flow behavior under the influence of Earth's gravity. It is the dynamic viscosity divided by density ρ (rho).

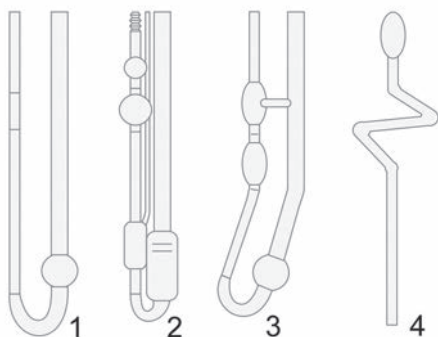
$$\nu = \frac{\eta}{\rho} \left[\frac{\text{m}^2}{\text{s}} \right]$$

Kinematic viscosity is widely established due to historical reasons: Gravity as the driving force does not require any elaborate technical equipment and is constant everywhere on Earth.

6.1: Laboratory Kinematic Viscometers

The most widely used technique for measuring kinematic viscosity is the use of a Gravimetric Capillary that is temperature controlled, usually 40 C for single grade oils, and both 40 and 100 C for multi-grade oils. Measurements using capillary viscometers are based on the relation between viscosity and time. The more viscous an oil, the longer it will take to flow through a capillary under the influence of gravity alone. There are several standardized capillaries in use today. Most laboratory instruments employ glass capillaries, or 'tubes.' A more recent advancement for field measure of kinematic viscosity

employs a split aluminum cell capillary. The instruments are designed to work as either direct-flow or reverse-flow capillaries. In direct-flow capillaries, the sample reservoir is located below the measuring marks. In reverse-flow types the reservoir sits above the marks. Reverse-flow capillaries allow the testing of opaque liquids and some can have a third measuring mark. Having three measuring marks provides two subsequent flow times and improves the measurement repeatability. Figure 1 shows different types of capillary tubes.

Figure 1: Common glass capillary types of kinematic viscometer.**GLASS CAPILLARY TYPES**

- | | |
|---------------|---|
| 1 – Ostwald | 3 – Cannon-Fenske |
| 2 – Ubbelohde | 4 – Houillon (Modified
Zeitfuchs crossarm) |

Manual constant temperature bath systems

These systems consist of a very precise temperature controlled bath, in which the direct flow capillaries are immersed. A sample of oil, usually 10 ml, is suctioned into the tube until it reaches the start point. The suction is then released and the oil flows by gravity through the controlled capillary section of the tube. Two or three marks are visible on the tube. An operator watches the meniscus of the oil as it passes the start point. At this point, the operator times how long it takes the oil to pass the final mark. The tubes are selected such that the test will take a minimum of 200 secs to complete. This makes it easier for manual timekeeping. ASTM D 445 is the method for kinematic viscosity and was originally written for the manual method.

The advantage of the manual system is that it is fairly inexpensive comparing to the automatic versions. Because of the requirement of minimum 200 seconds test time, it is fairly accurate. Different tubes are needed for oils of different viscosity ranges to maintain the test time requirement, and for such manual systems it is fairly easy to change the tubes.

The drawbacks of such systems are that the test is slow and labor intensive and after the test, tubes have to be cleaned manually.

Automated modified Ubbelohde method

A common system used by labs is an automated modified Ubbelohde method. A 10 ml bottle is placed in a small

carousel rack. The system draws oil up to the tubes as per the manual method, though in this case all the tasks are controlled by a computer program. The system does not require an operator to monitor and time the oil flow. With the automation the system maintains the accuracy of the manual system but removed labor for timing and tube cleaning. Systems can be equipped with dual solvent option for hard to clean sooty oil samples.

The downside of such systems is that it is still slow. For such system typically 12 samples per hour can be achieved with a 10 position carousel. The two tubes in the system are usually fixed in place so they are less flexible. It takes fair amount of solvent to clean the tubes (up to 15ml per sample) and 5 ml of oil sample per measurement.

Direct flow capillaries

These systems are preferred for in-service condition monitoring because they are more suitable for opaque fluids, and the lab versions have higher throughput and flexibility. Common names for this method are “Houillon” or “Hele-Shaw” technique. The ASTM method that describes this approach is ASTM D7279. A frequent question for anyone considering obtaining a viscometer is how this method compares to ASTM D 445, a far more widely known viscosity method. ASTM D 7279 has excellent repeatability, and to obtain identical ASTM D445 results, a standard offset (detailed in the method) is all that is required. For most users who are focused on the trend change, laboratory instruments designed using this technique have excellent accuracy and exceed machine condition monitoring requirements. To take a measurement using this technique a small sample of oil, between 0.6 to 1.6 ml, is pipetted and introduced directly into the tube that is heated to the desired temperature. Disposable pipette tips are used to minimize cross contamination.

One of the key advantages of direct flow systems is its high throughput, with each bath holding up to 4 tubes and all measurement in parallel, 25 to 45 samples per hour is easily achievable. Cleaning of the tubes are automated, and dual solvent options are available for hard to clean sooty samples. Figure 2 is a picture of Spectro Scientific SpectroVisc Q310 dual bath automatic viscometer.

**Figure 2:** Spectro Scientific SpectroVisc Q310 dual bath kinematic viscometer

6.2: Solvent-free Portable Viscometer

Introduction

This paper describes how using the hand-held, solvent-free SpectroVisc Q3000 Series device in the field provides immediate and accurate kinematic viscosity measurements, even when compared to traditional laboratory viscometers. The SpectroVisc Q3000 Series uses new solvent-free technology to accurately perform kinematic viscosity measurements requiring no calibration, no density verification, and no temperature measurement.

This discussion will first provide details on the SpectroVisc Q3000 Series' design and then describe how that design performs compared to traditional laboratory viscometers. Finally, a case study compares the measurement results between the SpectroVisc Q3000 Series portable, kinematic viscometer and the SpectroVisc capillary (Modified Zeitfuchs) tube viscometer used in many commercial oil analysis labs.

Traditional Viscometer Theory and Design

Current viscosity measurement techniques rely heavily on the use of capillary, cone and plate, and concentric cylinder viscometers. These devices are mainly limited to the laboratory setting and contain obstacles to portability. While the capillary viscometer suffers from difficult and lengthy procedures for calibration, cleaning, and temperature control, the rotational viscometer is hindered by its rotating parts and delicacy. Higher sensitivity viscometers have since been developed based on differential or light scattering methods, but these are expensive and laboratory based.

Some commercial instruments have been developed to address a need for portable viscosity measurement, especially where it is essential to determine the status of critical fluids in real-time. Such viscometers include attempts at miniaturization of the differential and rotational viscometers. Although these devices reduce sample volume, certain components remain complicated and costly, posing a challenge for their widespread adoption.

Other devices and methods have recently developed based on MEMS technology, including membrane oscillation frequency measurement, acoustic wave measurement, the piezoelectric actuated cantilever and the shear resonator. Despite requiring reduced sample volumes, many of these devices lack temperature control and are not kinematic in nature, so may not yield comparable results.

SpectroVisc Q3000 Series Viscometer Theory and Design

The SpectroVisc Q3000 Series viscometer design includes an upper sample loading well, microchannel, and temperature control electronics to measure fluids at a constant temperature of 40°C. Two models are currently available, the Q3000 which measures viscosity over the range 10-350 cSt, and the Q3050 viscometer with a range of 1-700 cSt. The SpectroVisc Q3050 viscometer also calculates oil viscosity at 100°C from the 40°C measurement with the input of the Viscosity Index for the fluid.

Operation of the device is simple; after loading ~60µl oil into the upper well of the chip, gravitational force causes the fluid sample to flow down the microchannel where a combination of emitters and detectors in the IR range detects its rate of progression. It requires no user calibration, temperature measurement, or density analysis.

This viscometer operates as a Hele-Shaw cell, where Stokes flow is present between two parallel plates. The distance between plates is necessarily small relative to the width and height of the plates. As depicted in the schematic diagram of Figure 5-1, the presence of only two parallel plates causes the micro fluidic device to be unbounded, meaning that the fluid is exposed to air on two sides.



Figure 5-1: Micro Fluidic Kinematic Viscometer - Schematic

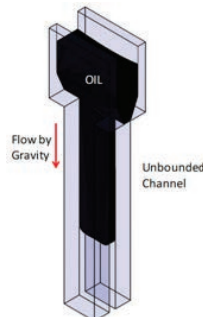
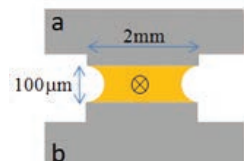


Figure 5-2: Concave Meniscus - Top View



The unbounded microchannel is very advantageous for cleaning; you just wipe the microchannel surfaces after separating the two parallel plates to clean the device. The optical detection method, where LEDs positioned on the one side of the microchannel and respective photodiodes on the other side are not obstructed by side walls, is also advantageous.

Although overflow of the microchannel might have been a problem based on the absence of side walls, surface tension generates a concave meniscus between oil and air, as seen in Figure 5-2. To have a positive pressure that forms this concave meniscus requires an oleophilic material.

The laminar flow condition dictated by the small gap between plates ensures the flow can be modeled as existing only in the vertical direction. At steady state under laminar flow conditions, viscous and gravitational forces are balanced such that where μ is dynamic viscosity, u is velocity, ρ is fluid density and g is gravitational acceleration. From that, the kinematic viscosity of the fluid can be determined using the average velocity, where U is the average velocity, g is the gravitational acceleration, and d is the channel depth. Here the dx^2 term is neglected because the geometry of the microchannel is straight and the fluid is moving due to only gravitational force. Near the funnel region this one dimensional equation is not valid due to transient effects of viscous forces balancing gravitational force. Regardless, these effects are avoided with placement of the optics sufficiently down the microchannel.

$$\mu \frac{d^2 u}{dy^2} + \rho g = 0 \quad v = \frac{gd^2}{12U}$$

To successfully operate the device as a Hele-Shaw cell depends on the aspect ratio of the microchannel being large enough. However, hydrostatic considerations must be considered due to the unbounded design. If the hydrostatic pressure by the oil exceeds the opposing pressure due to surface tension, the fluid will overflow through the unbounded sides. To maximize surface tension, aluminum acts as a microchannel material because it can be easily machined and forms a small contact angle with the investigated oils. For example, the contact angle between engine oil and aluminum surface is 2.73 degrees and engine oil surface tension is approximately 31 mN/m. The surface tension induced pressure value at the unbound surface is where R_1 is radius of meniscus (half of microchannel depth; 50 μm) and R_2 is infinite (the plate width in relative terms is very large).

It reasons that 620 Pa is the maximum hydrostatic pressure that the surface tension can hold when two aluminum

plates are 100 microns or 100 μm apart. Therefore, the maximum length of the microchannel is Based on the previous calculation, as well as the variety in surface tension and contact angle among oils, the microchannel length used in the Q3000 series viscometers is 42mm.

$$\Delta P = \gamma \left(\frac{1}{R_1} + \frac{1}{R_2} \right) \sim 620 \text{ Pa} \quad \Delta H = \frac{\Delta P}{\rho g} \sim 76 \text{ mm}$$

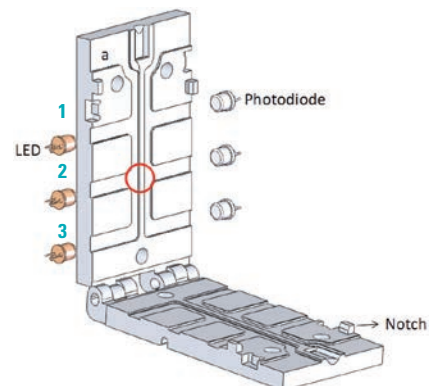
Figure 5-3 and 5-4 shows the two aluminum plates that were created by an ultra precision computer machining system and how they attach to a hinge that allows easy opening and closing.

The fluid passing between an LED and a photodiode causes a drop in the photodiode voltage. Using the time points that mark these voltage drops, the average velocity of the oil calculates from the elapsed time between photodiode 1 and 2 as well as photodiode 2 and 3. The average velocity is then used in Equation 2 to generate a kinematic viscosity for the measured sample. Two Resistance Temperature Detectors (RTDs) embedded within the aluminum plates enable a custom designed proportional-integral-derivative (PID) controller attached to a heating element to effectively maintain the temperature at 40°C.

Figure 5-3: Aluminum Plates with Hinge



Figure 5-4: LEDs and Photodiode Positions



Case Study – SpectroVisc Q3000 versus SpectroVisc Q300

Knowing the viscosity of a lubricant is critical for condition monitoring. As a result, field-based users need portable viscometers to immediately assess critical equipment while working on-site. Other portable viscometer options currently available as commercial products require solvents, density and temperature measurements to arrive at results. They do not correlate with laboratory viscometers, meaning that the collected data is not co-trended. In-use oils, in particular, make precise viscosity measurements by any technique a challenge given their particulates, water and combustion by-products.

The process of comparing results from one instrument to another involves factors, such as:

- Wide variability in the performance of in-use oils
- Variability in the performance of the viscometers
- Variability in application requirements

Rather than use a single benchmark to compare portable and laboratory viscometers, the following case study provides a method for determining whether a portable viscometer will “do the job” given particular requirements.

For this comparison, a SpectroVisc Q3000 serves as a portable viscometer and a SpectroVisc Q300 as a laboratory viscometer. The SpectroVisc Q3000 is a portable, solvent-free kinematic viscometer developed for applications where immediate results are required. The SpectroVisc Q300 is a capillary (Modified Zeitfuchs) tube viscometer designed for laboratory analysis.

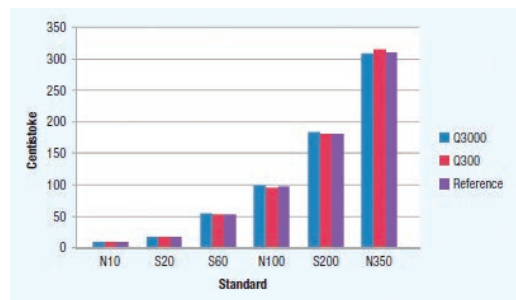
Both instruments measured the viscosity of a number of samples, and the measurements were compared from instrument to field viscometer. Two series of comparisons were made. The first set of samples consisted entirely of NIST-certified standards and the second sample set consisted of used oils.

Each sample was run three times, each on the SpectroVisc Q3000 and the SpectroVisc Q300 with kinematic viscosity readings taken at 40°C. Results from both sets were averaged and compared.

The application of NIST standards allows the accuracy of both approaches to be compared and also to see how closely the portable Q3000 reproduces results consistent with those of the laboratory. The used oil measurements, on the other hand, are for real-world, immediate results. Here the actual viscosities are unknown, making absolute accuracy comparisons impossible. The goal is to determine how the portable tool compares to the laboratory instrument across a spectrum of widely varying samples. If the results from the two approaches are “close enough” for the user, the portable tool can be substituted for the analytical instrument.

Figure 5-5 shows the performance of the Q3000 compared to the SpectroVisc Q300 over a range of certified viscosity standards. The Q3000 performs consistently across the calibrated range, with a relative standard deviation less than 2%.

Figure 5-5: Q3000 vs. Q300 Performance Using a Range of Certified Viscosity Standards



Results

The following data was obtained using a range of certified viscosity standards:

- Table 5-1 compares the Q3000 against the NIST references
- Table 5-2 compares the Q300 against the NIST references
- Table 5-3 compares results from the two solutions directly

Q3000	Ref	% Diff
10.13	10.03	1.00
17.93	18.04	0.61
54.86	54.08	1.44
99.26	97.15	2.17
185.00	180.80	2.32
308.67	310.90	0.72
Portable Viscometer		

Q300	Ref	% Diff
10.02	10.03	0.11
18.06	18.04	0.09
53.79	54.08	0.54
96.64	97.15	0.52
180.93	180.80	0.07
314.90	310.90	1.29
Lab Viscometer Q300		

Not surprisingly, the Q300 laboratory viscometer yielded results that are in line with referenced ASTM norms (0.44%) for variation. The portable Q3000 reported results were within its expected 3% specification, and the results from the Q3000 and the lab-based viscometer were also within 3% (Table 5-3). In the used oil comparison, Table 5-4 shows that results from the portable Q3000 were consistently within 3% of the bench top Q300.

Q3000	Q300	% Diff
10.13	10.02	1.11
17.93	18.06	0.70
54.86	53.79	2.00
99.26	96.64	2.71
185.00	180.93	2.25
308.67	314.90	1.98
Portable Q3000 vs. Lab Q300		

Sample	Q3000	Q300	% Diff
Used Engine Oil	54.56	55.24	1.22
Used Engine Oil	108.67	111.63	2.66
Used Engine Oil	132.00	128.80	2.48
Used Process Oil	158.33	161.80	0.98
Gear Oil	237.00	230.30	1.03

Table 5-4: This table compares used engine oils

Conclusion

The key question is whether 3% is adequate in the context of the user's specifications. As mentioned earlier, used oil can present significant challenges for repeatability measurements, especially with oil, water, fuel and particle contamination — any of which can drive repeatability above 5% between tests of the same sample. OEM engine and rotating equipment providers and users consider viscosity variations greater than 10% (from nominal values) to be the first evidence of potential problems. A portable viscometer can detect such issues immediately, allowing improved decision making and more efficient preventive maintenance.

Undoubtedly, the advent of new solvent-free technology for field-based viscosity monitoring offers the performance necessary to detect variations of in-use oil to signal an impending problem at the equipment site. In addition, case study results show agreement within 3% of all samples between traditional laboratory viscometers and the portable viscometers. This indicates that the SpectroVisc Q3000 Series device can deliver accurate results in a portable setting, using a fraction of the sample volumes required by commercial laboratory viscometers.

References

- [1] Huang, C.Y., et al., Viscosity and density measurements of macromolecules. *Angewandte Makromolekulare Chemie*, 1999. 265: p. 25-30.
- [2] van der Heyden, F.H.J., et al., A low hydraulic capacitance pressure sensor for integration with a micro viscosity detector. *Sensors and Actuators B-Chemical*, 2003. 92(1-2): p. 102-109.
- [3] Wang, W.C., P.G. Reinhall, and S. Yee, Fluid viscosity measurement using forward light scattering. *Measurement Science & Technology*, 1999. 10(4): p. 316-322.
- [4] Gilroy, E.L., et al., Viscosity of aqueous DNA solutions determined using dynamic light scattering. *Analyst*, 2011. 136(20): p. 4159-4163.
- [5] Faas, R.W., A Portable Rotational Viscometer for Field and Laboratory Analysis of Cohesive Sediment Suspensions. *Journal of Coastal Research*, 1990. 6(3): p. 735-738.
- [6] Kuenzi, S., et al., Contactless rotational concentric microviscometer. *Sensors and Actuators a-Physical*, 2011. 167(2): p. 194-203.
- [7] Sakai, K., T. Hirano, and M. Hosoda, Electromagnetically Spinning Sphere Viscometer. *Applied Physics Express*, 2010. 3(1).
- [8] Fitt, A.D., et al., A fractional differential equation for a MEMS viscometer used in the oil industry. *Journal of Computational and Applied Mathematics*, 2009. 229(2): p. 373-381.
- [9] Ronaldson, K.A., et al., Transversely oscillating MEMS viscometer: The "Spider". *International Journal of Thermophysics*, 2006. 27(6): p. 1677-1695.
- [10] Smith, P.D., R.C.D. Young, and C.R. Chatwin, A MEMS viscometer for unadulterated human blood. *Measurement*, 2010. 43(1): p. 144-151.
- [11] Choi, S., W. Moon, and G. Lim, A micro-machined viscosity-variation monitoring device using propagation of acoustic waves in microchannels. *Journal of Micromechanics and Microengineering*, 2010. 20(8).
- [12] Rezaadeh, G., et al., On the modeling of a piezoelectrically actuated microsensor for simultaneous measurement of fluids viscosity and density. *Measurement*, 2010. 43(10): p. 1516-1524.
- [13] Ballato, A., MEMS Fluid Viscosity Sensor. *IEEE Transactions on Ultrasonics Ferroelectrics and Frequency Control*, 2010. 57(3): p. 669-676.

Chapter 7: Fuel Dilution in Engine Oil

Introduction

Fuel contamination in oil is one of the most critical issues requiring high attention from reliability engineers. Engine oil samples are frequently monitored on site or by a remote lab checking fuel dilution levels. There are several ways of monitoring fuel contamination indirectly, using change of viscosity or flash point as an indication, or using very expensive instrumentation such as GC-MS or FTIR and complex data analysis algorithms.

There is need for a simple, inexpensive, and direct measurement tool to check the fuel contamination in the field directly, enabling reliability engineers to make decisions quickly on site or by the asset. Developed in collaboration with the U.S. Navy, the Q6000 Fuel Dilution Meter (FDM) fulfills the market need to provide simple and rapid determination of fuel dilution in engine oils. Chemical microsensors were first proposed as on-site and in-situ tools for engine and lubricant condition monitoring at the Joint Oil Analysis Program Conference in 1983. In the spring of 1990, the Naval Ship System Engineering Station (NAVSSSES) initiated a program to develop a portable SAW-based instrument to measure the fuel dilution in shipboard diesel engine lubricants. Microsensor Systems Inc. and the U.S. Navy combined their experience and expertise to design and build a small, rugged, reliable instrument, based on the SAW microsensors. After an extensive evaluation and testing period, the US Navy purchased over 100 Fuel Dilution Meters for use aboard ships. This instrument was the previous generation of the current Fuel Sniffer that applies exactly the same technology.

The Q6000 FDM provides direct, rapid and accurate measurements of fuel contamination in used engine oil. Because of its small size and simple user interface, it is ideal for field use or in the laboratory. The Q6000 requires no chemicals or consumables, making it inexpensive to use as well as fast and accurate. It is an ideal tool for maintenance professionals and laboratory

who are responsible for supporting automotive, railway, marine, and heavy equipment fleets. The focus of this paper is to provide details on how the Fuel Sniffer operates and how its method of operation compares to GC-MS (Gas Chromatography - Mass Spectrometry) and Flash Point methods. Finally, this paper presents two cases studies, one for the United States Air Force (USAF) that compares Fuel Sniffer methodology to GC-MS technique, and another case study which proves the Fuel Sniffer's ability to provide repeatable and accurate measurements among multiple samples.



Figure 1:
Q6000 Fuel
Dilution Meter

Accurate Fuel Dilution in the Field Using the Q6000 FDM: A Comparison to Gas Chromatography

Fuel contamination in a lubricant is one of the most important lubricant failure modes in internal combustion engines. The presence of fuel reduces the oil viscosity which decreases the load carrying ability of the lubricant. Unchecked fuel dilution issues in a lubricant can lead to severe wear to bearings, gears, and pistons. Other fuel dilution failure mechanisms include lubricant breakdown and seizure, increased oil oxidation and sludge deposits, as well as explosion due to the presence of light-end, highly volatile hydrocarbons.

The most widely accepted direct methods for analyzing fuel dilution in lubricants is by Gas Chromatography (GC) according to ASTM methods D3524, D3525, and more recently, D7593.¹⁻³ These methods provide highly accurate results and are designed for high volume labs due to the fact that they are expensive and require experienced technicians. Also, GC analysis can be very time consuming to get the best result. Many commercial laboratories have modified the ASTM methods in favor of a faster and more robust method developed by a leading GC manufacturer.⁴

Other conventional measurement techniques for fuel dilution are indirect measurements which include viscosity, flash point testing, and FT-IR spectroscopy. Viscosity tests

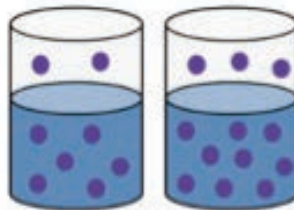
are routinely performed on a lubricant anyway, but a change in the viscosity does not exclusively conclude a fuel dilution problem. Flash point testing requires correlation tables generated from known samples in order to output fuel dilution in a useful unit, such as %. FT-IR spectroscopy cannot easily distinguish between fuel hydrocarbons and those present in the base oil making it difficult to achieve accurate results.

There is a need for a portable, on-site fuel dilution measurement. Obtaining results within a matter of minutes could mean the difference between a serious engine failure or a minor maintenance issue. The new Q6000 portable Fuel Dilution Meter (FDM) has been developed to meet this need.

Portable Fuel Dilution Measurement by the Q6000 FDM

The Q6000 FDM is a portable, battery operated fuel dilution meter that determines the concentration of fuel dilution present in an oil sample within a matter of minutes (Figure 1). The Q6000 FDM uses a unique patent pending fang design to pierce the cap of a disposable sample vial and draws in the headspace from the vial. The headspace flows over a SAW (Surface Acoustic Wave) sensor which reacts specifically to the presence of fuel vapor with a detection range of 0 – 15%

As per Henry's law, the amount of fuel diluted in the oil is directly proportional to the vapor in the headspace of a closed sample vessel at equilibrium (Figure 2).



The concentration of fuel vapor present in the headspace of a closed bottle at equilibrium is proportional to the amount of fuel present in solution.

Figure 2: Henry's law as it applies to fuel dilution.

Comparative Study

A comparative study between the GC reference method and the Q6000 FDM was performed. Three sets of typical lubricant calibration and test samples containing various amounts of diesel fuel dilution were obtained (Table 1). The calibration samples were used to calibrate the Q6000 FDM prior to analysis of the test samples. The samples were also sent to an ISO 17025 accredited commercial laboratory for comparative analysis to GC by modified ASTM D3524.1,4 The instrument used was a PE Clarus 500 gas chromatograph using a 7 point calibration curve (correlation coefficient of >0.999) created with mixtures of 15W-40 engine oil and #2 diesel fuel.

Sample	Type	N
A0	Calibration	New Mobil Delvac 1 ESP 5W-40 oil, no soot present, 5% diesel fuel dilution
A1	Test	Used Mobil Delvac 1 ESP 5W-40 oil, no soot present, unknown diesel fuel
A2	Test	Used Mobil Delvac 1 ESP 5W-40 oil, significant soot present, unknown diesel fuel dilution
A3	Test	Used Mobil Delvac 1 ESP 5W-40 oil, significant soot present, unknown diesel fuel dilution
B0	Calibration	New Esso Racing 20W-50 oil, no soot present, 5% diesel fuel present
B1	Test	Used Esso Racing 20W-50 oil, no soot present, diesel added to 13.4% fuel

Table 1: Diesel fuel dilution calibration standards and test samples used in the comparative study between the Q6000 FDM and GC methods.

The known samples A0 and B0 were used as calibration standards (prepared to 5.0%) for the Q6000 FDM. Calibration of the Q6000 FDM can be performed with any known sample within the detection range. To run the sample analysis, 0.5 mL of sample was dispensed using a graduated disposable pipette onto the felt disc inside the FDM sample vial. Samples were prepared in triplicate and analyzed consecutively (Figure 3).

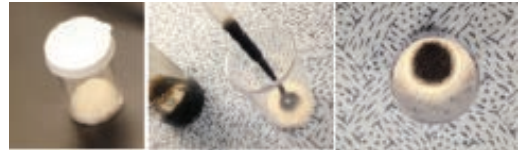


Figure 3: FDM sample vial and dispensing the oil sample onto the felt disc using a graduated disposable pipette.

Up to 5 samples can be prepared at a time for analysis on the Q6000 FDM. After the vials are capped, they must sit for one minute to equilibrate (per Henry's law) and then can be analyzed on the Q6000 FDM. The device is simple to operate relying on a touchscreen interface with display and audio instructions at each step. Results display within 2 minutes and the % fuel dilution found is displayed on the screen after each sample measurement (Figure 4).



Figure 4: Results screen on the Q6000 FDM showing the % fuel dilution found for a sample using a diesel calibration program.

The results from the comparative study are shown in Table 2.

% Diesel Fuel Dilution						
Sample	Oil Type	Condition	Q6000 FDM (% Diesel Fuel)			GC Analysis (% Diesel Fuel)
A1	Mobil Delvac 1 ESP 5W-40	Used, Not Sooty	1.8	1.9	1.9	1.98
A2	Mobil Delvac 1 ESP 5W-40	Used, 1.2% soot	0.3	0.2	0.4	0.22
A3	Mobil Delvac 1 ESP 5W-40	Used, 1.2% soot	1.8	1.9	1.9	1.80
B1	Esso Racing 20W-50	New oil, known 13.4%	13.9	13.1	13.0	12.95

Table 2: The % diesel fuel dilution results for each test sample.

The fuel dilution % of the known calibration samples as measured by the reference GC method of the known calibration samples were A0: 5.14%, B0: 4.97%.

The results for test samples A1, A2, and A3 on the Q6000 FDM correlate very well with the GC reference method, as shown, at about 0.2 and 2% diesel fuel dilution for both lightly used and very sooty samples. To test the higher end of the measurement range on the Q6000 FDM, a calibration standard of 5% (B0) and a known test sample of 13.4% diesel fuel (B1) in Esso Racing 20W-50 were prepared. The average result obtained on the Q6000 FDM for test sample B1 of 13.3% diesel fuel is accurate to the gravimetric value and also correlates well to the 12.95% obtained by the GC reference method.

The repeatability between all triplicate measurements for the Q6000 FDM gives high confidence in the result and the sampling method. The repeatability (typical) on the Q6000 FDM is <5% RSD. An operator can have confidence using this technology in the field trusting that it will correlate to the referee laboratory method.

The Q6000 FDM is much quicker and simpler to do accurate analyses on many different types of oils on-site. It only takes 2 minutes and just one known calibration standard in order to create a custom calibration on the Q6000.

Conclusion

The Q6000 Fuel Dilution Meter (FDM) is an easy-to-use, portable, solvent-free solution for detection of fuel dilution in oil samples. Analysis takes only a couple of minutes and accurate, repeatable results are obtained immediately. Compared to the most trusted laboratory method, gas chromatography, it is substantially less expensive, can be used by an operator without any special skill set, and provides faster results. The measurement is also easily adaptable by single point calibration to many different oil types.

References

- [1] ASTM Standard D3524-14. Standard Test Method for Diesel Fuel Diluent in Used Diesel Engine Oils by Gas Chromatography.
- [2] ASTM D3525-04(2010). Standard Test Method for Gasoline Diluent in Used Gasoline Engine Oils by Gas Chromatography.
- [3] ASTM D7593-13. Standard Test Method for Determination of Fuel Dilution for In-Service Engine Oils by Gas Chromatography.
- [4] Timothy D. Ruppel, Gerald Hall, and Andrew Tipler, "A Novel Method for High-Speed Determination of Fuel Diluents in Lubricating Oils," Field Application Report. Perkin Elmer, 2005, Pages 1—3.

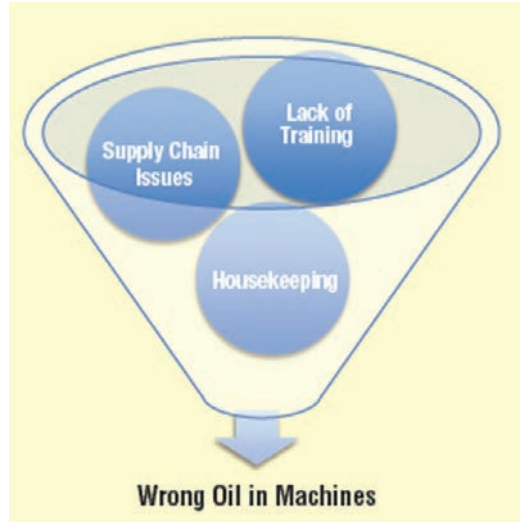
Section 3: Oil Analysis Practices and Success Stories

Chapter 8: Lubricant Mix Up – How to Catch it with Oil Analysis

Introduction

A recent survey with a local power plant revealed a lubricant mix up is the most common cause of lubrication-related problems. Any departure from the original equipment manufacturer's oil specification, specifically in terms in type and viscosity grade, will increase the probability of equipment failure and lead to increased costs in maintenance and repair operations. So why does it happen and how can it be prevented? This application note will review the causes of lubricant mix up and the oil analysis tests that notify the maintainer when a mix up has occurred.

No equipment owner or maintainer plans to put the incorrect oil in a machine. There are three major reasons why it occurs and these reasons, though not exhaustive, are universal across all organizations when this problem is encountered.



1. Lack of Training

Most mechanics and equipment maintainers (the personnel responsible for day-to-day machine maintenance) are not trained on what a lubricant does, and specifically what the differences in formulations mean. The only exposure they have is frequently a drum or pail label – which has limited information. Most identify lubricants based on viscosity grade only. Product sheets from lubricant suppliers are vague for competitive reasons, and petroleum marketers tend to emphasize the “our products cover your needs” message rather than specific details on what not to do. As a result, the “oil is oil” attitude is pervasive throughout the world, and the theory “better to lubricate with whatever oil is on hand than no oil at all” is used on a daily basis. Combined with this lack of knowledge are two other complications, rapidly increasing in recent years:

- a) The lubrication function is migrating from a traditional “maintenance function” to “daily operations function” and being increasingly managed by process operators who were not trained in maintenance or lubrication.
- b) The turnover in operator job positions is higher than maintenance specialists. As a result, training is lost regularly.

2. Supply Chain Issues

Lubricants are sometimes erroneously considered commodities by purchasing departments. In an effort to have a specification that is not brand specific, lubricant family names (many familiar sounding) are confused. If the product has the same viscosity grade and same function (e.g. Hydraulic AW fluid), it is assumed the oil can be added to the machine without degradation of performance. Additive compatibility, equipment sensitivity and corrosion side effects are not considered carefully, and lubricant suppliers may not have the opportunity to advise Purchasing of the importance of specific lubricants.

Many lubricant distributors perform TLM (total lubrication management) with specific responsibility for this area. It is incumbent on the customer or lubricant supplier partner to have inbound quality control to ensure the right oil is supplied.

3. Housekeeping

Poorly managed workshop areas and a disregard for lubricant integrity contributes to lubricant mixup. GMP practices and a defined quality function can minimize the mistakes made at this point. Color coded lubricant transfer jugs, labels and dispensers are also available.

All three of these areas will address the 5 Rs of lubrication: Right Type, Right Quality, Right Amount, Right Place and Right Time



If your lubricant storage area is like this you will have lube mixup issues

Role of Oil Analysis

Specific lubricant condition tests provide information about lubricant mix up. The tests are summarized in Table 11-1. Let's review some common tests used in condition monitoring and see how they can detect when the specified oil is incorrect for the application.

Table 11-1: Oil Analysis Tests

Test Method	Approach	Advantages	Limitations
Viscosity	Kinematic Viscosity	Can immediately tell if the wrong grade is present.	Small additions are within normal viscosity variations. Oils with different additive packs are not addressed.
Elemental Spectroscopy	ICP, RDE, XRF	Ash type additives easy to identify, and different additive elemental ratios can be easily observed.	Basestock mismatch not addressed. Ashless organic additives have not signaturred elementally.
Infrared Spectroscopy	FTIR, Direct IR	Functional groups and additive packages easy to detect, search and identify (FTIR). Ability to validate to a known oil (Direct IR).	Many basestocks/additive packages very similar and suppliers change formulations regularly. Difficult to be decisive on unknown sample
Gas Chromatography (Non-Routine)	GC, GC-MS	Different species elute at different times, particularly oils with clear ionization signatures; ability to measure .05% of lubrication mixup.	Compounded oils present challenging separation column selection. Several compounds coelute.

Viscosity

It is easy to spot when a lubricant of a different classification is present. For example, if a gearbox oil is tested and the result is 249 Cst @ 40C, and the expected viscosity range is 150, Cst +/- 20%, the most likely reason for the large departure is a higher grade oil was added. In-service oil is expected to vary no more than 20% from the normal viscosity.

Viscosity measurement is alone not sufficient if the mixed up lubricant added has the same nominal viscosity grade. For example, military aviation hydraulic systems call for both mineral and fire resistant hydraulic fluid. Both have exactly the same viscosity range and physical characteristics. In this case, the viscosity measurement is necessary but not sufficient for mix up detection.

Elemental Analysis

The presence of additive elements such as phosphorus, zinc, calcium, magnesium, barium and potassium, where prior trends indicated trace or no values, are a telltale sign of contamination. An increase in values or a change in the quantity or ratio of elements will often indicate lubricant mix up. A prior trend increases confidence in results especially when a change in magnitude is observed.

Elements	Current Sample	Previous Sample
Calcium	135	145
Phosphorus	1955	1386
Magnesium	80	100
Calcium	259	14
Zinc	780	24

Table 11-2: The current sample additive pack is significantly different pointing to lubricant mixing at top off

IR Analysis

Infrared spectroscopy is a recommended tool for detecting lubricant mix ups because it allows analysts to compare the molecular fingerprint of the unknown sample to a known reference (see below). Any unusual peaks can be quickly identified visually by superimposing a reference or prior spectra to the unknown sample. The most common methodology in laboratories is the Definitive Negative Test Method (i.e., the IR technician can confirm what it is not, rather than what it is). This is usually achieved with reference oil. A common request in the course of an investigation is to request a sample of the unknown and the many potential mix candidates. Superimposing these on the unknown oil will immediately identify differences in the oils. Performing any of the built in search/identify functions in the IR software will aid in the analysis.

Many molecules share similar IR vibrational frequencies.

FTIR is widely used in condition monitoring labs, and many oil analysis customers expect the technique to automatically alert when a lubricant mix up is in process. This is a false assumption.

FTIR condition monitoring tests, either the JOAP indexing or the ASTM methods, select specific areas of the spectrum where known degradation and liquid contamination is expected to be found. If the oil is mixed with another product, it is quite likely that the trend analysis result generated by routine analysis may not show a departure from trended degradation compounds in these areas, particularly if base stocks are the same. Mixing synthetic oils with mineral oil can cause variation, and it will only be triggered by the analyst if the integer value exceeds a slope alarm.

Direct IR techniques, used in FluidScan, are helpful if the environment and potential mix up is known beforehand and modeled. For example, military aircraft regularly are concerned with turbine and hydraulic fluid mix up – the aircraft performance is degraded if such an event occurs.

Specific “alien fluid” algorithms, tailored for the known contaminant, can be developed into a routine method – available on FluidScan (see below).

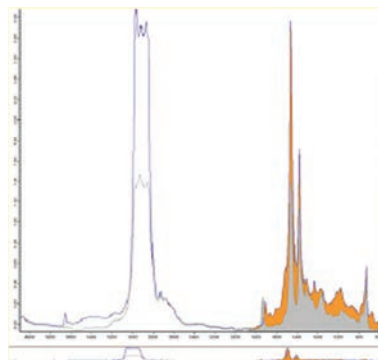


Figure 11-1:
Superimposed
IR Spectra

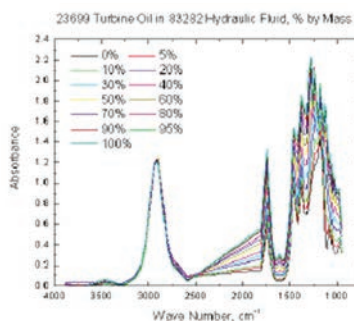


Figure 11-2:
Analysis of mix
up of oils to
determine
algorithms,
and report out
% w/w

The above tests are common oil analysis tests. They have benefits and limitations. IR remains a great screen test for lubricant mix up, and with more “known references” the more effective the test becomes. By routinely checking the fluid, metrics can be applied to improvement initiatives.

Chapter 9: Route-Based Oil Analysis

Benefits of Route-Based Oil Analysis

Personnel who carry out machinery health inspections at industrial plants today generally follow pre-defined inspection routes. This “walking-the-beat” approach has proven to be effective for analysis of vibration and infrared thermography because the information derived from on-the-spot, real-time inspections and data collection is extremely useful. These inspections are also economical in many situations where having permanently installed equipment on each asset would be too costly.

Until now, these pre-defined machinery health inspections have not included route-based oil analysis in order to determine lubricant properties quantitatively. It just was not possible previously to perform this type of analysis at each point along the route within a minute (or two at the most). Now, with the development of handheld tools such as the FluidScan® Q1100, it is feasible to obtain critical, quantitative oil parameters within a short time right at the sampling point (see Figure 12-1). As a result, it is possible to create and use an oil analysis route. The routes may be managed by Emerson AMS OilView software.

Route-based oil analysis is a big step beyond visual inspections by maintenance personnel as they go through a plant along a pre-determined route to perform greasing or topping-off of lubrication systems.

Of course, technicians should still observe the machinery visually while walking a route, but on-the-spot oil analysis can be done very quickly with good accuracy and with repeatable results. Walk-around analysis provides immediate feedback and the ability to retest right in the field, if needed. In many cases, it is not even necessary to use a sample bottle. Route-based oil analysis adds even greater value because the information provided from a structured database is always correct and consistent. No time is wasted, and no human error is incurred, since the route automatically associates reference information with each designated sample point.

By consistently identifying the correct reference oil and asset sample point with a test, the walk-around infrared spectrometer operator can achieve far better repeatability and accuracy than can typically be achieved in a commercial laboratory. Many commercial labs rely on secondhand information to consistently identify the exact lubricant and other pertinent information related to each test sample.

Today’s portable instruments used for route-based oil analysis are able to outperform lab instruments because the information available at the point of the test identifies the appropriate test methods, analysis parameters and



Figure 12-1: Route-based Oil Sampling

alarm limits for each lubricant sample on a sequential route. Simply stated, route-based oil analysis allows technicians to do a better job of oil analysis and get it done quickly because their instruments are programmed to always select the correct protocol and reference information for each point along the route.

Six elements must normally be addressed for a technician to conduct a route-based analysis with a portable analyzer in an industrial plant or around a fleet of mobile equipment (see Figure 12-3).

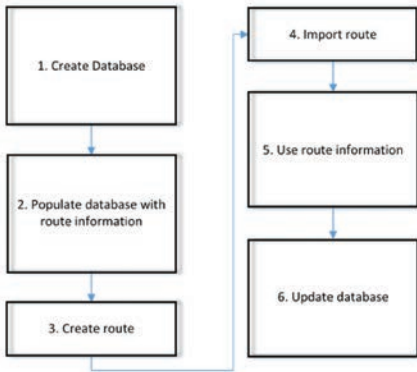
Figure 12-2: Route-based oil analysis observations

Observations	
0	Unit Usage
0	Oil Usage
0	Oil Added (Liters)
Unknown	Visible metal particles?
Unknown	Visible non-metal particles?
Unknown	Visible Process Material?
Unknown	Visible fibers?
Unknown	Water separated in sample?
Unknown	Milky from water?
Unknown	Visible varnish or sludge?

Back Save Change

1. Create a structured database in Emerson OilView.
2. Populate the database with route information associated with the oil and equipment in the database.
3. Select a preferred inspection route.
4. Transfer route information into a handheld analyzer.
5. Apply route information in the analyzer during in-field analysis.
6. Upload measurements and findings to the database.

Figure 12-3: Steps for creating a route and importing it to an in-field oil analyzer



Database Creation

A maintenance server is configured to generate oil analysis routes based on a set of analysis rules (see Figure 12-4). These rules may be based on time, a previously existing condition or specified by the maintenance engineer. The route could include the sequence, asset identification and/or reference oil information, such as oil name, type, property limits, etc.

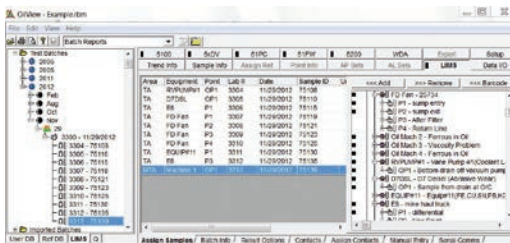


Figure 12-4: Creating a route using a structured database

Populating the Database

The generated route is synched between the maintenance server and the device to be used for the route-based analysis. This can be accomplished in a number of ways, including a straight database synch, a file download/transfer or a barcode list, which could serve the dual-purpose of a sample bottle label (see Figure 12-4). For a set of recurring routes, a binder could be constructed of 8½-by-11 sheets that could be pulled out and scanned whenever a given route is indicated (Figure 12-5).

Sample labels afford a benefit of potentially including other data independently collected from lab or other on-site test instruments into this same sample record. Still another way to accomplish this would be for the user to print and laminate labels and attach them to each sample point. Then, when the technician walks the route, he or she could scan the barcode to add that location to the route in the database.



Figure 12-5: A “route” of points represented by sequenced machine readable sample labels – generated from Emerson OilView

Route Analysis

With the route information downloaded into the handheld machine, lubricants are analyzed at each asset in sequence. The handheld unit operates in a self-guiding fashion so that the user is presented with the next asset's information when finished with the analysis of each asset. This may include GPS coordinates of the asset, a picture, serial number or simple instructions on where to find the next asset. The handheld device contains all the calculation tools necessary to perform the analysis.

The results of each lubricant scan are stored in the handheld unit. The technician can see the results as soon as they are produced. This can include alarm limits imported from the maintenance server so that the user can determine immediately if the machine is "in-spec," according to the analysis. If out-of-spec, additional instructions may be offered, such as collection of an oil sample at that machine for further analysis.

Transferring Data

When the route is complete, the handheld device is again synched with the maintenance server so the data can be uploaded and the database updated. The maintenance engineer can then review this data and use the server's analytical tools to determine in any further action is needed.

Planning a System

It is easy to envision how a route-based oil analysis system could be a valuable tool for a large facility where route-based machinery health monitoring is practiced. With the natural flow of data in a route-based system, decision-makers can see how on-the-spot oil analysis can be fit into the context of their other asset-management tools. It certainly provides a convenient and practical way to perform oil analysis with minimum or no paperwork. Furthermore, as at-line, in-line and on-line tools are developed for oil analysis as they have been for vibration monitoring, a similar path forward is envisioned for these two probes of machinery health.

By using route-based oil analysis, users can plan coverage for a whole range of assets based on economic considerations. Dedicated in-line and on-line analyzers that have the same capabilities as the handheld at-line tools may be used where appropriate, with that data stream tied together with the route-based system at the maintenance server and overall system.

It is clear that oil analysis and other probes of machinery health, such as vibration and thermography, have fundamental differences that must be considered when planning how and when to use emerging oil analysis tools. Oil analysis, which is inherently a physical and chemical investigation of the oil including base, additives, contaminants and wear debris, is significantly different than vibration or thermography, both of which use information arising directly from the machinery itself.

When the lubricant within that machinery is analyzed for chemical information, users have another means of assessing the health of the machine, the type and extent of system contamination, and the functional condition of the oil. This extra step needed for oil analysis helps to explain why oil analysis instrumentation is still emerging for on-the-spot, full and quantitative machinery assessment. Nevertheless, significant recent advances in the speed, size and weight of quantitative oil analysis tools indicate route-based oil analysis paradigm can now be a reality.

Chapter 10: Oil Analysis for Mining Equipment Maintenance

A low-grade surface mining operation depends upon huge and expensive equipment such as Hitachi 5500 shovels and Caterpillar 793 haul trucks. Oil analysis is essential to identify problems so they can be corrected before they cause equipment to go down for major repairs. In the past, the mine sent samples to an outside lab for analysis but the cost of this approach limited the number of samples that could be analyzed. In addition, the four day leadtime to receive the results created the risk that damage could occur before the results were obtained.

The mine overcame this problem by creating an in-house lab based on the Spectroil Q100 rotating disk electrode (RDE) atomic emission spectrometer and SpectroFTIR Q400 oil analyzer from Spectro Inc.

The mine can now obtain oil analysis results in only 12 minutes and the reduced cost per sample has made it possible to increase the number of samples analyzed by nearly a factor of 10. In its first year of operation, the in-house lab has identified savings of over a million dollars in downtime and over a million dollars in repairs, with several of the larger incidents generating savings that completely paid for the purchase price of the instruments.

Key role played by heavy equipment

The mine relies heavily upon enormous equipment designed to quickly and efficiently move massive amounts of ore. A typical example is the Hitachi 5500 excavating shovel which has a maximum payload of 280 tons and is powered by a 2600 horsepower engine. A Caterpillar 793 haul truck weighs 270,000 pounds and can haul 250 tons of ore. This truck is powered by a 16 cylinder, 2478 horsepower engine. These large pieces of equipment are worth approximately \$15,000 per hour when operating, so minimizing downtime is critical to profitability. Repair costs are also enormous with, for example, the cost of rebuilding an engine running about \$400,000. Even more important, the sudden failure of a major system in heavy equipment, such as a truck or shovel, could create a safety issue.



Figure 1: The Spectroil Q100.

Value of oil analysis

Oil analysis has long been an important part of maximizing uptime and minimizing repair and replacement costs at the mine as well as the vast majority of other major mines in the world. Oil analysis determines the amount of various metals in the oil, providing a fast and inexpensive way to gauge the amount of wear in the machinery. Oil analysis also helps determine the condition of the oil such as by measuring solids formed by oil oxidation and the viscosity of the oil. Tracking the condition of the oil helps reduce the risk of catastrophic failure and can also reduce the high cost of changing and disposing of oil in heavy machinery. It takes only a few minutes to take an oil sample and the analysis cost is low so oil analysis is a simple and practical way to keep a mine operating profitably.

In the past, the mine performed some analysis itself with a very basic analysis tool but its lack of reliability meant that the company primarily used an outside lab. The outside lab provided reliable results but it took two days to ship samples to the lab using Federal Express and another two days for the lab to analyze the samples. As a result, it was not uncommon that, by the time the mine received information indicating a problem, the equipment had already failed. This situation resulted in downtime and repair costs substantially higher than would have been incurred if the repairs had been made a few days earlier. Another weakness of the old approach is that the outside lab charged approximately \$12 per sample and it cost another \$15 for shipping costs. These costs limited the mine to analyzing an average of five samples per day.

Establishing an in-house lab

“I got management approval to establish an in-house lab and spent over a year looking at different technologies and brands,” said the reliability centered maintenance (RCM) coordinator for the mine. “I concluded that Spectro stands far above the rest both in performance and cost of their instruments. For example with other instruments, the sample has to be pumped through the machine and flushed out, creating the potential for cross-contamination. The Q100, on the other hand, has a disposable wheel that avoids this potential problem.”

The mine uses the Spectroil Q100 to analyze wear, additives and contaminants in mineral or synthetic, petroleum-based products and also uses the SpectroFTIR Q400 to measure oil degradation and contamination. The mine is also planning to purchase a SpectroVISC Q300,

semi-automatic kinematic viscometer and SpectroT2FM Q500 analytical ferrography laboratory in the near future. The mine also uses the SpectroTrack laboratory information management system (LIMS) to automatically capture analysis results from the instruments and store them in a database where they can be accessed over the Internet and used to track trends. When a lab tech emails the RCM coordinator to alert him of a possible problem, he immediately logs into SpectroTrack and checks the history of the machine and other similar machines to help determine the best course of action.



Figure 2: The T2FM Q500.

Savings in downtime and repair costs

The in-house lab has already identified four problems, each of which would have cost more to fix than the entire cost of outfitting the in-house lab if the mine had waited for an outside lab to identify them. In one case, the in-house lab identified a glycol leak in a Caterpillar 793 haul truck. "If we had waited for results from an outside lab we would have ruined the engine which would have cost \$400,000 to rebuild," the RCM coordinator said. "Instead we fixed the leak at a cost of a few hundred dollars. On the same truck a few months later we saw the tin and lead levels in the oil start to rise, indicating that the bearings were starting to go. Putting in new bearings avoided the catastrophic failure that was about to occur and saved \$164,000, the value of the engine minus the cost of putting in the new bearings. Rebearing cost only about 12 hours of downtime compared to 88 hours if we had had to change the engine, saving another \$1,140,000."

In another case the lab detected a level of copper in the oil of the transmission on a Caterpillar 992 loader so high that the core would have been ruined in another eight hours of operation. The transmission had to be rebuilt but preserving the core saved \$78,000. Oil analysis can also sometimes save warranty costs. When one engine was rebuilt the rebuilder pulled a sample from the bottom of pan and found high levels of glycol so they blamed the failure on a glycol leak which would not have been covered by the warranty. "I sent the sample to our in-house lab and they said there was no glycol," the RCM coordinator said "For confirmation, I sent another sample

to an outside lab and they also said there was no glycol. So the manufacturer covered the cost of rebuilding the engine which saved us about \$150,000."

"For every engine we save, we pay for the cost of operating the lab for an entire year, including the depreciation on the instruments," the RCM coordinator concluded. "We have already saved four engines in 18 months of operating the in-house lab, not to mention many other cases where we have had smaller but still substantial savings. The in-house lab is superior not only in providing faster results but also in providing results at a lower cost – about 12 cents per sample – which has made it possible to increase the number of samples we are able to analyze to about 50 per day. Finally, we are also saving a substantial amount of money on oil changes because the analyzers tell us exactly when an oil change is needed. With hydraulic shovels that use 1500 gallons of oil at a cost of \$3 per gallon, these savings add up. All in all, in-house oil analysis has provided substantial savings in downtime, equipment repair costs and oil costs that have paid back its cost several times over in only 18 months."

For more information, contact Spectro Scientific, Inc.
Ph: 978-431-1120 Web: www.spectrosci.com

Chapter 11: Oil Analysis for Servicing On-the-Road Transportation Vehicles

Kitsap Transit is a public transportation agency, based in Bremerton, Washington and serving the county of Kitsap, which operates routed bus service, foot ferry service and service for elderly and disabled passengers. The agency has long been concerned with preventing engine failures and optimizing maintenance schedules for its fleet of approximately 362 pieces of equipment including transit buses, cutaway small buses, vans and three passenger only ferries.

Previously, the maintenance department monitored the engines of vehicles in its fleet by sending oil samples to an outside laboratory. The analyses provided measurements of heavy metal content, fuel and radiator fluid contamination, etc. The problems with this approach were cost and time: it cost \$40 per sample using a thirdparty lab, and it took 14 to 21 days to receive the results. The cost limited the number of samples that were analyzed and the lead time created the possibility that a failure might occur before the results were received.

Kitsap Transit purchased an OSA4 TruckCheck® automated oil analysis system from Spectro Scientific in 2014. The system enables the maintenance team to perform on-site analysis of oil samples previously done in the lab including Total Base Number (TBN) results for less than \$1 per sample in less than 15 minutes.

“Bringing oil analysis in house made it possible to begin analyzing all of our bus engines on a regular basis,” said Dennis Griffey, maintenance supervisor with Kitsap Transit. “Within the first few months, the analyzer identified two engines that were close to failure. We saved about \$40,000 by eliminating the need for an out-of-frame rebuild in one case and by getting the rebuild done by the dealer as part of the warranty in the other.”

Kitsap Transit agency operates 43 bus routes

The agency operates 43 bus routes and ferry service from Bremerton to Port Orchard, Washington. Kitsap Transit’s ACCESS service provides door-to-door and curb-to-curb service for the elderly and disabled. It runs 32 worker/driver commuter routes operating to and from the Puget Sound Naval Shipyard and Naval Base Kitsap/Bremerton in which the drivers are full-time shipyard employees. The agency’s fleet includes nine 2012 Arboc, 17 2010 Arboc, 2 2003 EIDorado, 45 Gillig models from the 2000s

and 42 MCI worker/driver coaches from the 1990s, 50 e-350 and e-450s, 11 g-4500s in ACCESS service, 124 vanpool and vanlink vans and various cars and trucks.



Figure 1: Kitsap Transit has approximately 150 buses in its fleet.

“We have long been aware of the potential of oil analysis to maximize uptime and minimize repair and replacement costs, but we were missing out on much of its benefits due to the fact that we only were able to do infrequent spot checks on our engine oil,” Griffey said. Oil analysis determines the amount of various metals in the oil, making it possible to gauge the amount of internal wear. Oil analysis also helps determine the condition of the oil by measuring solids formed by oxidation and by determining the viscosity of the oil. Tracking the condition of the oil helps reduce the risk of catastrophic failure and it can also reduce the high cost of changing and disposing of oil in heavy machinery.

The agency operates on a fleet schedule, which involves performing an in-frame rebuild every 300,000 miles. In an inframe rebuild, the block is rebuilt without removing the block from the frame. This type of rebuild typically involves replacing the pistons, rods and rings but not the spinning parts. An out-offrame rebuild requires removing the block from the frame and replacing the camshaft, crankshaft, valve lifters, pistons, rods and rings. The cost of an in-frame rebuild ranges from \$13,000 to \$25,000 for the buses operated by Kitsap Transit and an out-offrame rebuild is roughly double the cost of an in-frame rebuild. An in-frame rebuild is sufficient in most cases; however, an out-of-frame rebuild often becomes necessary when an engine failure occurs.

Technicians trained to use oil analyzer in two hours

"When we saw the automated TruckCheck oil analysis system at a trade show, we were surprised at how reasonable the cost was," Griffey said. "We figured that we could pay for the instrument if we were able to avoid the need for just two out-of-frame rebuilds by preventing engine failures. I presented a cost justification to the department director and gained approval to include it in the budget. Each of our technicians took a two hour course, which was sufficient to enable them to operate the instrument and perform oil analysis."

The TruckCheck analyzer is a tandem spectrometer that integrates an Optical Emission Spectrometer (OES) and an infrared module. The OES excites a portion of the used oil sample and measures the concentration of sub-microscopic metals in solution. These metals are present due to normal and abnormal component wear. The infrared module scans a portion of the used oil sample to measure the physical properties of the oil and look for contaminants. An onboard computer controls both spectrometers and tabulates the results gathered from each, including information about the oil and the vehicle as provided by the operator. The software generates a report that includes the test results and a diagnostic statement that alerts the operator if any of the results are outside normal ranges.

Kitsap Transit technicians now perform oil analysis routinely whenever they change the oil of a bus engine. "One of the first things we discovered was that our oil was not meeting the V40 or V100 spec even right after we changed it," said Bill Rich, lead mechanic for Kitsap Transit. "Furthermore, we found that we were losing an additional 10 points on the V40 spec over the life of the oil. Running our engines with the viscosity low tends to increase engine wear. So we switched to a different oil supplier. Now we monitor the oil with every change to make sure it meets the spec." The V40 spec refers to the viscosity at 40°C while V100 refers to viscosity at 100°C.



Figure 2: Technicians obtain oil analysis results in less than 15 minutes.

\$15,000 saved by avoiding out-of-frame overhaul

"A month ago I tested a mid-size Cummins diesel engine and found that it was high on heavy metals," Rich said. "The engine was not scheduled to be rebuilt for 11 months so it's highly likely that it would have failed some time before that point. In that case, we probably would have lost the spinning components and the engine would most likely have required an out-of-frame overhaul which would have added about \$15,000 to the cost. By detecting the problem, we saved about half the cost of the analyzer."

In another application, a 40 foot bus with a Detroit Diesel Series 60 engine was brought in for maintenance because the oil was overfull. "In the past, oil analysis would probably not have been performed," Rich said. "But now, we routinely run oil analysis on any engine brought back into the shop. In this case, the analysis showed that the overfill was caused by diesel fuel infiltrating the oil. The engine almost certainly would have failed and it was approaching expiration of the warranty, so we would have been responsible for the \$25,000 cost of an out-of-frame overhaul. Instead, we sent the results to the dealer and they fixed the problem at no cost to the agency."

The TruckCheck analyzer also detected heavy fuel dilution indicating premature wear on several engines used in small cutaway buses. The engines would have failed at some point and it is likely that by the time they failed, the engines would have been out of warranty and Kitsap Transit would have been responsible for an out-of-frame overhaul. Kitsap Transit is now working with the bus dealer to resolve the issue, which will be covered by the warranty. The cost of doing an out-of-frame overhaul on these engines is about \$25,000 so the potential savings are significant.

"We originally justified the purchase of the automated analyzer based on our bus engines but we have since found many other uses for it," Griffey said. "For example, a bus was brought in recently with low power steering fluid. The technician analyzed the steering fluid and found high amounts of iron. We traced the iron to the pump and replaced it, which solved the problem. We have also started using the analyzer to check the engine oil in our three ferries. With only three ships in our fleet, even having one ship out of service is a major problem. We have not yet seen any problems but we are more confident about our ferry service knowing that we will be able to detect any issues long before they threaten to take a ship out of action. Finally, we are preparing to begin analyzing the transmission fluid of our fleet, which we believe will provide additional savings. Overall, the analyzer has already paid for itself in just a few months and we are continually finding new areas where it can save money and improve utilization of our fleet."

Chapter 12: Oil Analysis for Offshore Drilling Reliability Service

A new approach

It is hard to think of another environment where an equipment failure could have a greater negative impact on employee safety and lost production than an offshore oil and gas production platform. Yet offshore platforms are among the world's most difficult locations to operate and maintain equipment. In most cases, skilled people and supplies can only reach the platform by ship or helicopter so the cost of bringing technical specialists, replacement equipment, spare parts and tools to the platform is high. On nearly every offshore platform, oil analysis plays a critical role by alerting the maintenance team to problems that may damage a vital system. An effective oil analysis program also helps to efficiently allocate scarce resources by planning maintenance based on equipment condition rather than time intervals. Most offshore platforms take oil samples and ship them by helicopter to onshore labs for analysis. Once the results are available, they are sent back to the platform.

The problem with this approach is it might take a week for the sample to reach the lab, another week for the lab to perform the analysis, another week to process the results and get them back to platform, and perhaps one more week before technicians view the results and take action. By the time the results are acted upon, the equipment may have failed. This could put personnel at risk and cause production outages or require costly shipments of replacement parts.

A new approach involves flying a technician with a portable oil analysis tool to the rig. The technician samples all of the equipment on the rig and provides instantaneous feedback. Immediate maintenance actions can be taken to avoid equipment failure. The operator can also determine the equipment which requires non-urgent maintenance or the assets that do not require any maintenance. This information allows valuable on-platform resources to be used for other duties.

Value of oil analysis on offshore platforms

It costs more than \$1 billion to operate a typical production platform over its 10 to 20-year life cycle, so operating costs per day can be estimated at \$100,000 to \$300,000. A typical offshore platform contains millions of dollars of machinery that is critical to the crew's safety and whose failure can easily put the platform out of operation. Oil analysis has long been the gold standard in the offshore industry for monitoring the condition of

critical equipment. Oil analysis determines the amount of various metals in the oil, providing a fast and inexpensive way to gauge the amount of wear in the machinery. Oil analysis also helps determine the condition of the oil by measuring solids formed by oxidation and by measuring the viscosity. Monitoring oil condition reduces the risk of catastrophic failure and can also reduce the high cost of changing and disposing of oil in heavy machinery.

As with other activities, oil analysis is much more difficult to perform offshore than onshore. The crew operating the platform typically does not have the time or equipment needed to perform oil analysis themselves. They collect samples from the various rotating equipment on the platform such as flooded screw compressors, turbo gas powered generators, fire water pumps, diesel engines, gearboxes, pumps, crane engines, hydraulic systems, etc. They label the samples, put them on the helicopter for shipment to the onshore lab and wait for the results. If any of the samples were mislabeled the results may be inaccurate. When a positive result is received, technicians must take immediate action before validating the results because it would take too long to analyze another sample from the same piece of equipment.

Moving from onshore to offshore oil analysis

One major oil producer, now using this approach, sent an oil sample to a lab and approximately one month later received results that indicated a problem with an air compressor. Unfortunately, the compressor failed the day before the results were received and the company had to incur the cost of replacing the machine. The oil producer asked John H. Carter Inc. to travel to its platforms with a portable oil analyzer and provide immediate oil analysis results. We researched the available portable oil analysis instruments and selected the Spectro Q5800 portable analyzer. The Q5800 is comprised of the following four modules:

- Infrared spectrometer with flip top cell design; tests for Total Acid Number/Total Base Number, water content, soot, oxidation and new fluid validation.
- Kinematic viscometer determines viscosity without solvent and with a low sample volume.
- Filtration Particle Quantifier (FPQ) provides solvent-free particle counting to less than 4 $\mu\text{m}/\text{ml}$.
- Elemental analysis module performs wear metal and sand/dirt analysis to identify abnormal wear and contamination ingress using X-ray Fluorescence (XRF) technology.



Figure 1: The Spectro Q5800 portable analyzer

The oil producer then contracted with John H. Carter Inc. to provide on-site oil analysis at its offshore platforms in the Gulf of Mexico. A Carter technician flies by helicopter to offshore oil platforms with a portable instrument and performs the same tests offered by full-service laboratories. The portable instrument provides the same accuracy as full-size laboratory instruments yet it fits into a backpack and is easily transported on a small helicopter. The all-in-one unit provides immediate oil analysis results. If there is an abnormal result, the platform personnel can act immediately to resolve the problem. Another key advantage of the portable analyzer is the ability to immediately re-check every positive result to ensure the initial test results are accurate. The retesting prevents false positives and allows substantial cost savings by avoiding unnecessary repairs or replacements.

Examples where offshore oil analysis saved time and money

John H. Carter Inc. has been providing on-site oil analysis to an oil producer for a year. There have already been several cases where the savings from on-site oil analysis exceeded the full year's cost of the service.

1. For example, technicians on one platform replaced the diesel engine on a crane. When the technician visited the platform and tested the oil, the viscosity was 70 when it should have been 120. The technician ran additional tests and discovered the presence of diesel fuel in the oil. A mechanic put dye in the fuel supply and found a broken injector line was leaking diesel fuel

into the oil sump. This leak had the potential to damage the engine or even cause a fire. The oil analysis results made it possible to fix the problem with a low cost solution – replacement of the injector line.

2. In another case, the oil analysis results on a large gas turbine compressor showed a high metal particle count. The technician queried the platform's maintenance team and discovered they had recently replaced a valve in the lube oil system. The technician wondered whether the oil particle count might have spiked in response to this maintenance, so he flushed the lube system and ran another test. This time the test showed a much lower particle count although still above normal values. After discussing the situation with the maintenance foreman, the decision was made to do nothing immediately but to retest the equipment the following month. When the equipment was retested, the particle count had returned to normal levels. According to the maintenance foreman, if the oil had been tested by an onshore lab, there would have been no chance to do an immediate follow-up study. It would have been necessary to, at the minimum, perform vibration testing and possibly perform even more expensive repairs.

Data management and reporting

The results of offshore oil analysis are uploaded to the Spectro Scientific SpectroTrack information management system. The results are available not only to the maintenance team on the platform but to onshore managers and analysts who track trends and provide recommendations on whether or not to invest in a certain piece of equipment.

Conclusion

The current oil analysis method used on most offshore platforms takes up to a month to send samples to a lab and receive the results. On-site oil analysis has the potential to provide major value and benefits by allowing testing on the rig and delivering immediate answers to the maintenance team. Faster results can prevent breakdowns and avoid unnecessary maintenance. The savings are realized by the ability to get answers quickly and better manage equipment uptime on the offshore rig.

For more information, contact Spectro Scientific, One Executive Drive, Suite 101, Chelmsford, MA 01824. Ph: 978-486-0123 Web: www.spectrosci.com

Chapter 13: Oil Analysis for Municipal Water Treatment Maintenance

Oil analysis helps a water plant cut annual maintenance costs by \$25,000

The city of Chandler (Ariz.) Municipal Utilities Department (MUD) employs coagulation, sedimentation, active flow control and filtration to produce 60 million gal per day (mgd) of water at its surface water treatment plant. In addition, the system includes 27 wells and 21 booster stations controlled by SCADA. Water distribution is provided through more than 1,200 miles of water main, in excess of 7,500 fire hydrants and more than 25,500 water valves. The city also has several facilities that treat wastewater, and in some cases, return the purified water into the aquifer through injection wells. The city's state-of-the-art reverse osmosis facility treats a 1.25-mgd wastewater stream produced by local industries.

The water production division runs deep well pumps, vertical turbine booster pumps, flocculators, flocculant collectors, and other pumps and mixers. The city relies heavily on this equipment to provide Chandler residents and businesses with a sufficient and continuous supply of potable water.

The city of Chandler previously changed oil in all motors, gearboxes and pumps in its water treatment facility twice a year at an approximate annual cost of \$50,000.

"In order to prevent damage to critical equipment, oil was changed every six months according to the equipment's maintenance instructions," said John Ardans, water systems maintenance superintendent for the city of Chandler MUD.

The maintenance staff recognized that the cost was high, but could not determine which oil changes could be deferred without damaging critical equipment. "We

recognized that much of the labor and materials involved were wasted, but at that point we had no way to know when the oil actually needed to be changed," Ardans said. "So we had no choice but to change it."

Evaluating Alternatives

Hoping to reduce unnecessary expenses, the city initially purchased a multi-parameter oil analyzer— which measures conductivity, resistivity and dielectric loss— and began using it in an effort to more accurately target oil changes.

"Unfortunately, our first oil analyzer provided inconsistent results," Ardans said. "When we checked it by testing the same sample twice, the results were all over the map. So we did not feel confident about relying on these measurements to safeguard our mission-critical equipment. Another weakness of the analyzer was that we needed to provide a new sample of oil with every sample of oil to be measured, which was inconvenient."

Management also considered using an outside testing firm to test the oil of the water plant's equipment. The annual cost of performing quarterly testing was estimated at \$15,488; the annual cost of biannual testing was estimated at \$7,744. Outside testing offered the potential for significant savings, compared with changing oil based strictly on schedule. A limitation of outside testing, however, is that it typically takes about two weeks to receive test results, so the potential for identifying a problem and correcting it immediately is reduced.

Searching for Solutions

Ardans decided to look for another analyzer. He identified Spectro Scientific's FluidScan Q1000 portable fluid condition monitor and was impressed by its direct infrared spectroscopy (DIR) approach, which provides a more accurate alternative to conventional total acid number (TAN)/total base number (TBN) analysis. DIR operates without wet chemistry and requires no solvents and only one drop of oil for analysis. This approach provides direct quantitative measurement of a lubricant's condition. The analyzer immediately detects lubricant contamination, and degradation and cross contamination in mineral and synthetic engines, including gear, hydraulic, turbine and transmission oils, as well as biodiesel and diesel blends. It analyzes for TAN, TBN, oxidation, nitration, sulfation, additive depletion and incorrect (or mixup) lubricant within water content, soot and glycerin in addition to fatty acid methyl esters in biodiesels.

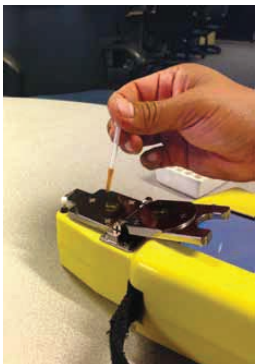


Figure 1: Only one drop of oil is necessary for analysis.



Figure 2: Booster pump

Justifying the Purchase

Ardans proposed that the treatment plant purchase the FluidScan Q1000 analyzer. To justify the purchase to management, he estimated that the annual costs of internally testing the water supply equipment would run about \$5,109.28 per quarter for labor and supplies. Assuming that the new analyzer would reduce the oil change frequency to once per year or less provides a savings of \$25,000 per year, ensuring return on investment within a year. Ardans also estimated that the analyzers would save \$10,378.72, compared with quarterly testing using an outside testing service. In addition, the analyzer provides immediate results, compared with the two-week wait for results from an outside testing service.

Management approved the purchase of the FluidScan Q1000 analyzer and city of Chandler MUD personnel began using it to perform quarterly checks on the health of 200+ pieces of critical equipment.

"We found both the accuracy and repeatability of the instrument to be excellent," Ardans said. "At this point, it appears that we will be able to increase the average time between oil changes to one year or more for all of our equipment. At the same time, we will be checking oil on a quarterly basis, so we are much more likely to prevent a premature failure than in the past. Simply saving one piece of equipment would more than pay for the analyzer instantly."

The city also purchased SpectroTrack software, which interfaces with Spectro Scientific instruments and keeps track of asset information relating to service intervals, maintenance actions, locations, status, etc. Users can see a comprehensive, historical view of fluid condition for one piece of equipment, a department or the entire plant. The software provides a single end-to-end view of a sample lifecycle from sample submission and receipt to results entry. The software also provides all trending, imaging, numerical and textual asset data in one secure location.

Cost Savings

With the successful rollout of oil analysis in the water supply division, the city is planning to begin analyzing oil samples from an additional 200 pieces of equipment in its wastewater treatment division. The savings in this area are expected to be approximately equal to those achieved on the water supply side. The city of Chandler also is planning to hire a predictive maintenance technician whose responsibilities will include collecting oil samples for both the water supply and wastewater treatment divisions. This person's responsibilities also will include performing vibration analysis, acoustical analysis and thermography to identify other issues that might put equipment into danger.

"Condition-based predictive maintenance is fast becoming the standard for forward-looking facilities that want to maintain the highest availability of their equipment, while minimizing unnecessary maintenance," Ardans said. "It is a proven tool to reduce operating costs while preserving the environment. The FluidScan gives us near real-time results at a fraction of the cost of a commercial lab. Changing to a condition-based model should allow us to optimize drain intervals and save enough money to pay for this tool in about a year."

Chapter 14: Oil Analysis for Industrial Plant Reliability Management

Introduction

This section provides you with three case studies which prove that industrial plant on-site lubrication programs yield outstanding results in terms of return on investment and short payback times. These programs help you cut costs, especially maintenance costs that go straight to your bottom line.

By developing an on-site industrial plant lubrication program based on analysis versus calendar year, oil test results are better and oil refurbishment is even better yet. These programs assure in-service lubricating and hydraulic fluids are kept clean, dry, and usable - and that the fluids are correct for the applications they serve.

For example, the lubrication program at an automotive assembly plant reported more than 700% return on investment with a 2 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 three principle areas: less fluids consumed, less reactive maintenance, and more deferred maintenance.

Most industrial applications do not stress the oil to the point where it is soon degraded. However, heavy-duty diesel engines, high temperature application, and harsh chemical environments are exceptions. The challenge for these applications is to:

- Monitor/reduce system contamination
- Monitor/reduce mechanical wear

In most cases, simply knowing when and how to properly filter oil can bring it back to a condition that is better than new. This information and subsequent action can easily reduce your oil consumption to a fraction of what it is today. As a bonus, you will dramatically increase your machinery reliability.

Therefore, your complete industrial plant lubrication program should contain the following processes:

- On-site oil analysis for screening of all new oil
- Organized storage and distribution of lubricants
- On-site oil analysis to determine oil condition and monitoring results
- Portable off-line filtration to return oil to a clean state

Pulp and Paper Industry Case Study

A large paper mill in the southeastern United States narrowly averted the failure of a wood chipper that could have cost the company as much as \$100,000 in repairs and lost production time. Fragile babbitt bearings guiding the chipper shaft were fragmenting, possibly due to a slight misalignment or imbalance, and the wood yard supervisor was not aware of their condition. Fortunately, a recently initiated on-site oil analysis program identified the problem, leading to the prevention of a catastrophic failure.

The chipper had been receiving what was considered adequate lubrication – a quarterly oil change along with filtration. However, calendar-based lubrication is often not a satisfactory solution, especially in dirty, dusty areas where oil can become contaminated very quickly, creating big problems before the next scheduled change. Many pulp and paper mills are finding that machine lubrication is far more effective and even less expensive when lubricant changes are based on their condition, which can be determined through a program of periodic sampling and oil analysis.

Rather than adding to the cost of maintenance, a lubrication management program (including sampling and analysis) can actually save a substantial amount of money in a large mill, which could be spending \$250,000/ year or more on lubricants. A mill of this size could reasonably expect to reduce oil consumption by 20% (\$50,000) by changing lube oils based on their condition rather than the calendar. The mill could actually save more than three times this amount, or \$150,000/year, by reducing the annual purchase, handling, storing, or disposal of 20% of its lubricants.

On-site labs make good sense for most pulp and paper mills, which generally have more than 100 oil systems. The mill gets better control over sampling, and testing can be done as often as necessary. The results are available immediately, so a retest can be done very quickly, if needed.

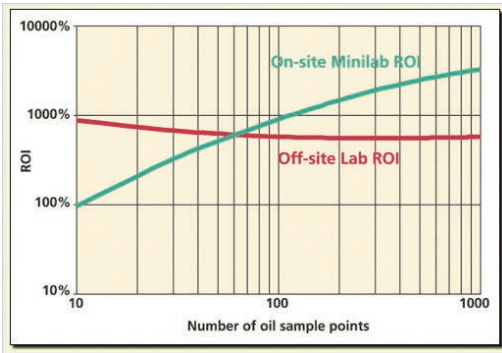
Mike Lawson at the Bowater Paper Mill in Calhoun, TN, would rather test oil at \$15 per sample than replace the 35 gallons in a gearbox, the total cost of which would be \$480. That includes \$140 for the oil, \$240 for two mechanics working six hours, \$50 to dispose of the used oil, and another \$50 to restock. The ROI in this case would be \$480/\$15, which equals 3200 percent!

The Spectro 5200 Trivector Minilab was installed at the paper mill to sample and test the oil before the decision was made to simply change it. Test results confirmed that the oil was not degraded and was actually quite clean. Changing this oil was not needed and may have introduced unwanted contamination.

The following graph displays how an on-site minilab, such as the Spectro 5200 Trivector Minilab is equally effective as pricier independent testing laboratories, demonstrating a lower per-sample cost and resulting in a better return on investment (ROI), as Figure 15-1 shows.

In conclusion, any paper mill that collects and tests fewer than 50 lube oil samples per month is missing problems that are costing far more in labor (and other expenses) than the cost of a more expansive program. It takes about one week a month to collect and conduct on-site testing of 100 samples from critically important equipment. The payoff in both labor and cost savings is far greater than the time spent doing this work.

Figure 15-1: Return on investment (ROI)



Steam Power Plant Case Study

Similar to the Bowater Paper Mill, power plant industries can greatly benefit from on-site lubrication programs as well. For example, the Gorgas Steam Plant on the Warrior River near Birmingham, Alabama chose to monitor oil condition in-house to avoid particularly dangerous events that could occur if one of its gearboxes fails.

Because most of the plant's units are from the '50s, '60s and '70s, lubrication contamination is a large issue. A good lubrication program that includes on-site oil analysis enables you to trend data on your equipment.

This capability provides for a rapid response, if needed, and can potentially save significant dollars. When combined with other technologies, it can help a power plant to operate optimally.

To that end, the plant now uses the Spectro 5200 Trivector Machinery Health Oil Analyzer. Its microscope attachment checks for wear debris - critical to the plant's needs.

Trouble began during a routine monthly oil analysis. Ferrous readings climbed daily from zero to 352 to 443. The previous month's analysis showed a zero ferrous reading, indicating a problem loomed. After draining the gearbox and examining the internals, they found that the thrust bearing had deteriorated to the extent that gearbox failure was imminent. In addition, the 10-B pulverizer would not have made it into peak season.

The bearing was easily replaced and two months after the initial discovery of ferrous content, the replacement was complete. The oil was re-sampled and cleared with a green light.

Here's how the cost savings add up. When Alabama Power had experienced a similar problem, prior to its use of an on-site lab, a catastrophic failure of the gearbox required a total rebuild with a cost of over \$400,000. However, the ability to stop this failure before it occurred, using on-site analysis, the total cost incurred was only \$30,000 - \$20,000 for the bearing and \$10,000 for the installation.



Rominserv Plant Case Study

Rominserv is a Romanian company which integrates industrial services, maintenance, project management and technical upgrades. The company maintains a Predictive Maintenance Laboratory where the vibration and lube oil data is processed and evaluated in order to predict when a machine will need to receive service in order to maintain expected levels of performance.

Only producing 10,500 tons/day, the company set a goal to operate at full capacity by 2009. In addition to this increased production, the company needed to reduce unnecessary maintenance work orders and costs. They also wanted to improve overall quality to meet the worldwide export regulation standards and increase Overall Equipment Efficiency (OEE).

When Rominserv decided to implement a predictive maintenance program based on condition monitoring, they chose Emerson's CSI 2130 Machinery Health™ Analyzer interfacing with AMS™ Suite: Machinery Health Manager to support this program. To access a complete picture of the health of the mechanical assets, Rominserv used Spectro's 5200 Trivector Machinery Health Oil Analyzer to ensure the quality of oil in order to extend the monitored equipment's life.

Program Milestones

- January 2005 – first vibration data acquisition using CSI 2130
- September 2005 – first lube oil analysis with the Spectro 5200 Trivector Analyzer
- By February 2006, the database for Petromedia platform (Rominserv's parent company) contained approximately 1,000 pieces of rotating equipment;
- Currently, there are over 1,200 dynamic pieces of equipment monitored using the AMS Machinery Manager (electric motors, pumps, gear boxes, fans, steam turbines, centrifugal and reciprocating compressors).



Romniserv Case in Point

#1 - Vibration Analysis Results

By analyzing the diagnostics, the company could pinpoint both critical equipment issues and those issues due to the equipment process. Through vibration monitoring, the company was able to detect the type of failure and the exact location of the damage.

Such was the case when an early break of the safety bolt on the rotor of a critical pump was detected. The fault could have resulted in a sudden pump breakdown that would have shut down an entire unit. However, the problem was discovered in time, and the flow was shifted to another pump without incident. The faulty pump was removed and repaired without incurring extensive damage. The estimated cost of lost production due to an unplanned shutdown of that unit would have been at least \$2.8 million USD.

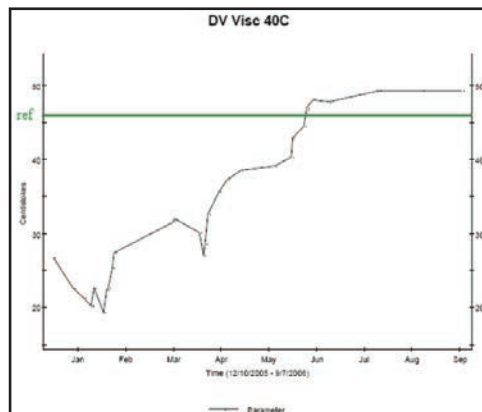
Romniserv Case in Point

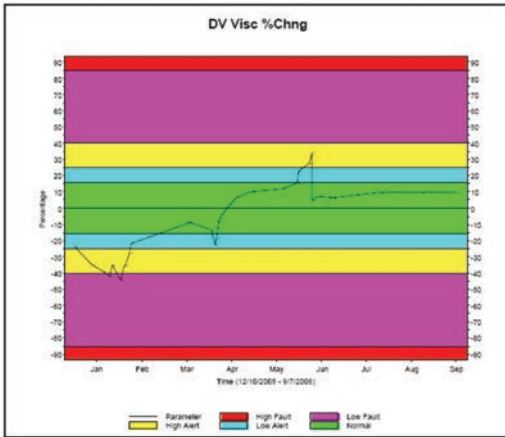
#2 - Lube Oil Results

By periodically sampling the lubricating oil in 150 critical machines and analyzing those samples with the Spectro Q5200 Trivector, factors other than vibration can be identified that can degrade the performance of dynamic operating equipment. Dirt and debris, water in the oil, and other contamination leads to the early recognition that internal damage may be occurring.

In this case, through oil analysis, Romniserv avoided a complete shutdown by detecting a severe lubrication problem in a critical machine. It is estimated that the economic benefit is \$7.3 million USD.

Using the Spectro Q5200 Trivector, the company started by first analyzing just the oil viscosity. In the graphs below, kinematical viscosity was 27cSt@40C and the KV percent change at alarm level is between blue code and yellow code.





A third analysis adding centrifugal action and with the kinematic viscosity at 20cSt@40C yielded the percent change at maximum admissible levels (mauve code). In addition, the water and particle quantity was within normal limits.

Figure 15-2: Trivector Plot

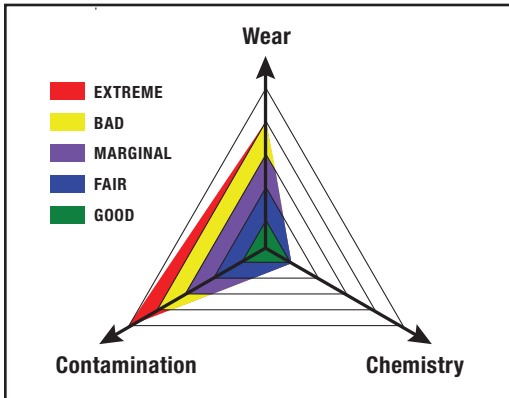


Table 1: Root Cause Failure Analysis

The root cause failure analysis looks like this:	
Causes	Actions
Low Oil Viscosity	Isolating the degassing tank return pipe
Malfunction of degassing unit	Eliminating the oil from the de-gasser
Water in oil	Adding new oil, but with higher viscosity
Steam and oil labyrinths high clearance	Periodical centrifugal actions

Actual costs of restoring lubrication:	
Costs	Value (USD)
New Oil	\$9,141
Contaminated oil (transport and commissioning)	\$627
Installing bleeding oil pipe from degasser (materials and labor)	\$370
Oil analysis	\$992
Total	\$11,130
Complete shutdown costs:	
Costs	Value (USD)
Total shutdown, production	\$7,151,210
Repairs, estimated materials and labor	\$133,160
Complete shutdown, estimated cost	\$7,284,370

In addition, the number of repairs decreased due to:

- Successfully applying predictive maintenance programs using new techniques
- Increasing quality of repairs and spare parts
- Employee awareness concerning importance of quality maintenance

Romniserv achieved the following business results:

- Saved an estimated \$10.1M by avoiding unnecessary downtime
- Over 10 times recovery of investment
- 35% to 45% decreased downtime for repaired equipment
- Potential production capacity increase of 20% to 25% by 2009
- Saved 25% to 30% on equipment spending

Conclusion

As the case studies demonstrate, industrial plants can benefit significantly by having a good on-site oil analysis program with condition based oil maintenance in place. This type of program will monitor your oil supply to make sure the oil is clean, dry and fit for use. In addition, wear debris analysis alone is your indication that surface damage is taking place within your machinery. Knowing these indicators early and up front, is your ticket to a industrial plant that contains healthy oil - your most precious commodity.

References

Portions of the content of this chapter are extracted from the following sources:

- [1] C.C.Ilie, Rominserv, Bucharest, Romania, www.rominserv.com
- [2] Ray Garvey, Emerson Process Management, "Prevent Mill Downtime by Knowing The Condition of Your Lubricants"
- [3] Ray Garvey, Emerson Process Management, "Outstanding ROI When Industrial Plant Lubrication Programs Are Supported by International Standards"

Chapter 15: Oil Analysis for Reliability Service

Component

Oil analysis detects changes in lubricant chemistry, wear particles that indicate component defects, and contaminants from outside the lubrication system. “The Spectro 5200 Trivector Analyzer has proven its ability to identify principle root causes of equipment failure such

as unseen corrosion, contaminants, improper lubrication and water intrusion,” said Niels Maes, Asset Health Analyst for Allied Reliability Group. “Early identification of these problems enables preventive intervention prior to catastrophic failure.” This article describes a number of typical examples of how oil analysis can reduce repair costs and improve throughput.

Sample Date	Oil Ref	7/7/2014	4/14/2014	1/7/2014	11/19/2013	10/15/2013	10/10/2013	10/4/2013	8/9/2013	7/10/2013	5/15/2013	3/1/2013
Sample #	(Click Here!)	9450	9306	9024	8881	8842	8811	8773	8630	8610	8558	8441
Lab #												
Analyst		Coservic	Coservic	Frederik	Kenny	Kenny	Kenny	Kenny		Kenny	Koen	Koen
Unique ID		9450	9306	9024	8881	8842	8811	8773	8630	8610	8558	8441
Wear Status		0	0	0	0	54	0	100	0	100	100	0
Cont Status		30	41	41	14	77	30	100	21	100	100	41
Chem Status		0	0	0	0	0	0	100	0	78	90	0
Unit Usage												
Oil Usage												
Oil Added												
NonFe Idx		8										
PC Vol < 6u		30	3.87	1.81	0.94	1.81	3.45	4.14	0	2.93	0	0.58
PC Vol 6-14u		31	4.95	6.81	8.07	1.42	7.16	4.04	0.75	2.42	1.01	0.75
PC Vol >14u		32	0.17	0.43	0.4	0.23	1.39	1.77	1,983.15	0.26	1,971.24	1,523.34
PC Vol Total		11	8.99	9.05	9.41	3.47	12	9.94	1,983.90	5.61	1,972.26	1,524.09
System Debris		33	0.22	0.23	0.24	0.09	0.3	0.25	49.6	0.14	49.31	38.1
Visc 40C	150	26										
Visc 100C	18.8	27										
Visc Idx	142	28										
Chemical Idx		0	-6.3	-0.7	1.5	-1.9		-1.8	999	-0.3	76.5	165.5
Contam Idx		1	0	0	0	0	65.5	0	999	0	999	999
Dielectric	2.13	2	2.07	2.12	2.14	2.11	2.12	2.11	80	2.13	2.89	3.78
Ferrous Idx		3	0	0	0	0	65.7	0	999	0	999	999
% Water		4	0	0	0	0	0.066	0	999	0	999	999
LCont Droplet		5	0	0	0	0	4	0	2	0	2	4
LCont Ferrous		6	0	0	0	0	0	0	1	0	1	1
LCont NonFe		7	0	0	0	0	0	0	3	0	3	0
DV Visc 40C	150	9	149	148	140	143	147	146		148	178	159
DV Visc %Chng		10	0	0	-6	-4	-2	-2		0	19	6
Cnts <4		12	88,925	60,712	46,338	37,307	86,615	87,874	31,089	61,368	30,468	24,217
Cnts >6		13	25,269	32,266	31,782	6,349	30,748	18,249	31,567	11,794	30,820	24,556
Cnts >10		14	558	1,185	2,857	346	2,021	1,025	31,320	369	30,320	24,232
Cnts >14		15	60	105	156	69	373	318	30,567	58	29,359	23,502
Cnts >18		16	14	32	23	23	105	155	29,751	21	28,236	22,715
Cnts >22		17	5	13	6	9	42	88	28,957	10	27,145	21,969
Cnts >26		18	2	7	3	3	21	48	27,497	5	25,140	20,650
Cnts >32		19	0	3	1	1	8	20	25,582	2	22,585	18,940
Cnts >38		20	0	1	0	0	3	5	22,690	1	18,741	16,482
Cnts >70		21	0	0.1	0	0	0.2	0	13,997.30	0	8,491.10	9,617.80
ISO >4		22	24	23	23	22	24	24	22	23	22	22
ISO >6		23	22	22	22	20	22	21	22	21	22	22
ISO >14		24	13	14	14	13	16	15	22	13	22	22
NAS 1638		25	13	14	14	11	14	13	18	12	18	18
pH		29										

Table 1: Oil Analysis shows water intrusion problems in can filling machines

Eliminating Water Intrusion in the Gearbox and Oil Tanks

A line fills up to 50,000 cans per hour, and then seams the filled cans. The filling machines have an “Automated Clean in Place” program that flushes them with large volumes of water and chemicals. ARG monitors the main oil reservoirs with oil analysis. Over a period of five months, the main drive showed recurring problems with water intruding into the gearbox and oil tanks. The oil was changed frequently and the seals were inspected and replaced but this did not solve the problem. Allied Reliability Group suggested supplying dry air to the oil reservoirs. The overpressure was limited to a small level to avoid blowing out the seals. This approach eliminated the water intrusion problems.

The same factory uses a bottle washing machine that rinses bottles before they are filled. Gearboxes drive the translation chain which moves bottles through the machine. Vibration measurements have been successfully used for condition monitoring of the ingoing shaft which operates at 55 rpm. Comparatively, the outgoing shaft operates at only 1 rpm, much too slow for accurate vibration measurements. Therefore, oil analysis is performed on the shaft lubrication reservoir every three months.

A recent measurement using the 5200 Minilab showed a sudden increase of wear particles in the oil. The first step was to change the oil and re-run the oil analysis, but the level of wear particles was also high in the new analysis. The number and size of wear particles continued to increase, revealing particles larger than 40 microns. The machine was shut down and inspected at the first planned stop. The inspection revealed a severely damaged bearing on the outgoing shaft.

The problem was solved by overhauling the reduction gearbox. If the problem had not been detected by oil analysis, the gearbox would have crashed and caused other parts to fail. Production would have been lost because the line could not operate the rinsing machine until the repairs were complete.

A labeling machine in the same factory has the ability to run at 60,000 bottles per hour. The high complexity and structural limitations of this machine means that certain parts can only be monitored by oil analysis. Oil analysis of the gearbox reservoir revealed an increased viscosity, a large increase in the dielectric constant, and a water intrusion.

An inspection of the gearbox showed a large amount of glue intruded into the oil reservoir because a seal failed. If the problem had not been identified by oil analysis, the gearbox would eventually have stopped, resulting in high repair costs and production losses. Accurate oil analysis provided relatively inexpensive corrections that fixed the

problem during the next scheduled production stop.

A steel mill uses a cold rolling process in which metal stock passes through one or more pairs of rolls to reduce its thickness and thickness variations. The skin pass is the final cold rolling pass and involves the least amount of thickness reduction, typically 0.5% to 1%. The skin pass also produces a smooth, uniform surface on the rolled steel plate. The numerous reducers which drive the skin pass are lubricated from a shared oil reservoir containing 6,000 liters (1585 gallons) of Shell Omala 220 oil.

A recent oil sample showed an increased ferrous index, increased number of particles, and a drop in viscosity from 220 cSt to 82 cSt. Allied Reliability Group advised the steel mill to urgently inspect the oil reservoir. The inspection showed that several heating elements failed which caused the particle count and ferrous index to increase. The inspection also revealed a failure in the circulation pump which caused the oil to overheat and reach high local temperatures. The steel plant replaced the broken heating elements, repaired the circulation pumps, and replaced the oil during a previously planned outage. A new oil sample taken from the same point revealed that the problem was solved. If the company had continued to operate the machine with bad oil the gearboxes would have been damaged, resulting in increased repair expenses and lost production due to an unplanned outage.

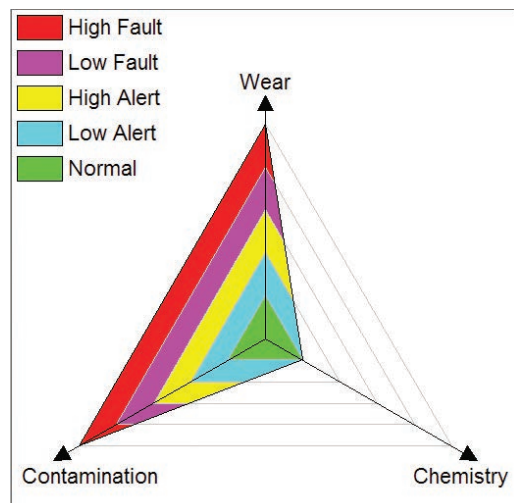


Figure 1: Oil analysis detects problems in slow moving shaft

The vibrations of faster-moving shafts in the same steel plant are monitored using vibration measurements. But a shaft that drives a gearbox at 9 rpm moves too slow for vibration measurement to be effective. Instead, an oil sample is analyzed every three months.

The most recent oil sample showed an increase in particle count and ferrous particles. Vibration analysis on other shafts driven by the same gearbox did not show any anomalies. The gearbox was opened up and inspected at the next planned stop. The rolling elements of the slow moving axle showed clear signs of grinding. The outer ring of the bearing was black due to the extreme heat generated by the grinding. The bearing would have obviously crashed if it had not been replaced. Oil analysis in this application saved a considerable amount of money in reduced repair costs and by avoiding a production outage.

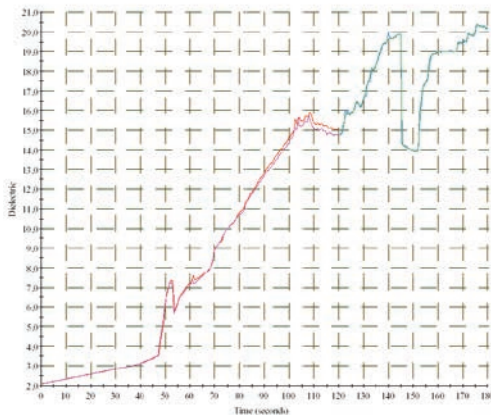


Figure 2: Oil analysis detects problem in fan balancing system

A steel producing plant uses a 3 meter diameter fan that turns at 1000 rpm to remove gases from steel converters. The gases are so hot that the fan blades need to be cooled with water. This causes steel dust to adhere to the fan blades, causing them to go out of balance. To compensate for this imbalance, four oil chambers are integrated into the fan, arrayed at 90 degrees from each other around the diameter of the fan. Oil is pumped between the chambers in order to balance the fan. Oil analysis is performed on a regular basis in this balancing system.

At a certain point a large increase in the water and contaminant level was detected in the oil. The problem was immediately reported to the maintenance team which planned an inspection at the first scheduled stop. A couple of days after the analysis was performed and before the inspection, the oil pump crashed due to the

high level of water. An inspection showed a problem with one of the seals, permitting cooling water from the fan blades to enter the balancing system.

An oil sample retrieved from a piston compressor showed a high level of water and the presence of ferrous particles. Based on these results, the compressor was overhauled. During this inspection, the camshafts indicated high levels of wear. The wear was presumably caused by reducing the lubricating properties of the oil caused by the water intrusion. The water intrusion was in turn caused by a failure of the water separator. In this application, oil analysis reduced repair costs and prevented a production outage.

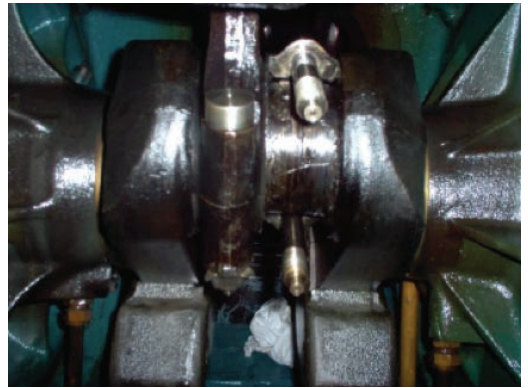


Figure 3: Oil analysis identified problem in piston compressor

“These applications are typical of the savings in repair costs and lost production that can be provided by oil analysis,” Maes concluded. “Asset health monitoring finds defects early to prevent unexpected failures and help the organization plan and schedule maintenance. The 5200 Minilab provides clear indications of metal wear, lubricant chemistry and contamination. The instrument also generates machinery health trend analysis to support data driven predictive and preventive maintenance decisions. Lubricant condition monitoring also conserves oil and reduces oil disposal costs by driving maintenance on an asneeded basis.”

Chapter 16: Oil Analysis for Engine Generator Maintenance

Portable Oil Analysis Tools Reduce Routine Maintenance Costs

What do a power plant, a hospital, a police station, and a remote mine have in common? They all have essential assets requiring uninterrupted power, commonly powered by an engine generator as primary or backup power. Engine generators, often termed “gensets,” combine an electrical generator and an engine. They supply electrical power where normal utility power is not readily available or is unstable. Gensets are used for temporary power demands and are often mounted on trailers or transportable skids.

Unlike large facilities that typically have on-site central oil analysis labs, smaller, temporary, and backup generation has traditionally depended on preventive, time-based oil maintenance. However, now, portable, handheld oil analysis tools are widely available and can be used to extend oil drain intervals and reduce routine costs for these generation assets. These tools are getting a boost with the recently amended U.S. Environmental Protection Agency (EPA) National Emission Standards for Hazardous Air Pollutants (NESHAP) rules for emergency backup gensets. The new rules allow condition-based oil drain intervals, so asset owners can realize the benefits of oil analysis. This article outlines the challenges and solutions available to portable/emergency genset owners who have previously incurred the cost of time-based oil changes.

Routine Maintenance and Oil Condition

Some of the main operating costs of running and maintaining large engine generators are the material and labor costs associated with changing oil based on a fixed operating time interval. This routine is often recommended by the engine manufacturer and increasingly by local regulations aimed at curbing emissions. Oil changes are suggested based on operating hour or calendar-based intervals, regardless of whether the generator has been running at full load or is idle for most of the time. Until now, this task was nonnegotiable, especially if the genset was under warranty. The U.S. EPA actually mandates oil changes for stationary engines used for emergency backup power.

Here are some issues with scheduled oil changes that trouble engine owners:

- Good oil gets changed unnecessarily. Not all generators run at the same load; therefore, it is likely that an oil change is unnecessary for some generators at the recommended change interval. This causes increased operating expense and waste, including material, labor, service engineer utilization, efficiency, as well as recycling cost. If an oil change interval can be extended for generators, the cost savings can be significant.
- Scheduled oil changes will not solve an ongoing contamination problem. Engine damage due to contamination of the lubricant can continue, and usually increases in severity.
- Catastrophic failures can still happen, and the cost of repair and downtime is not insignificant, even though it might be infrequent.

The Role of Oil Analysis

Forward-thinking genset owners and service providers have recognized these issues for some time, and they employ off-site or on-site oil analysis to determine the lubricant and equipment condition. In turn, they can determine if the oil can be extended or if the genset requires an overhaul.

The U.S. EPA has now acknowledged the benefits of condition-based oil changes founded on oil analysis results. The agency recently amended its regulations for stationary generators in emergency or backup mode to allow for extended changes if oil condition condemnation limits are not exceeded (Table 1).

Table 1: Time’s up! These are the new condemnation limits for in-service oil. *Source: U.S. Environmental Protection Agency*

Parameter	Condemning Limits
Total base number (compression ignition reciprocating internal combustion engines only)	<30% of the total base number of the oil when new
Total acid number (spark ignition reciprocating internal combustion engines only)	Increases by more than 3.0 milligrams of potassium hydroxide per gram from total acid number of the oil when new
Viscosity	Changed by more than 20% from the viscosity of the oil when new
% water content by volume	>0.5

The rule specifically states that condemned oil must be changed within two days of the engine owner receiving information that oil has exceeded any of the specified limits. If oil condition is examined at the time of scheduled service, a decision can immediately be made as to whether the oil needs to be changed or if minor repairs are needed. This approach reduces both operation and maintenance costs, and the engine runs longer.

A similar situation can occur in managing an automotive fleet. Time-based oil change has been proven to generate additional waste due to unnecessary oil changes. Though the cost savings are real and the marketplace is starting to support them, the question is why condition-based oil changes aren't a popular practice.

One reason is that the investment in a dedicated laboratory is not always practical. In mining operations and large power generation plants, it is common to have central oil analysis labs located on-site to continuously monitor the oil and machine condition of equipment. Decisions about oil change and other maintenance activities are made based on the recommendation of experienced laboratory data analysts.

While this is a very good industrial practice, it is difficult to apply this practice in the case of engine generator fleets because of the large, upfront capital investment, as well as the need to hire laboratory technicians and data analysts. Even if a central laboratory is established, the distributed or temporary nature of the gensets prevents service engineers from making immediate decisions due to the delay in getting results from the central lab. This is the problem with relying on contract labs to perform such work.

Another reason is that previous technologies for on-site oil analysis are insufficient to implement an effective condition-based oil change practice. The tools used to monitor oil condition need to meet the following requirements:

- Easy to use. There's no need to hire an experienced oil expert.
- Portable. Maintenance engineers can carry it from one generator to another.
- Fast. Engineers can use their on-site time more efficiently.
- No waste stream and no recycling of hazardous material chemicals. This minimizes the cost of training to handle, store, transport, and recycle chemicals.
- Comprehensive. The tool should capture the complete picture of oil condition with minimal chance of false alarms.
- Repeatable and definitive. Decisions can be easily made.
- Cost-effective. Return on investment is one or two years.

As you can see, this is not an easy list of requirements. There are many tools on the market that can partially meet them. The tools may be simple and easy to use, but not definitive, or they may be accurate, but expensive, difficult to use, or hard to deploy in the field.

Recently, Spectro Scientific introduced a comprehensive set of portable oil condition analyzers that provide a complete picture of in-service oil condition. Each tool is battery powered, small in a handheld form, and as accurate as laboratory instruments. These portable tools are even being used in some oil analysis labs.

Each tool uses a small oil sample—measured in drops—and does not generate any waste stream. No chemicals are needed to analyze the oil, so no hazardous materials or recycling are needed. Without sample preparation, it only takes a few minutes to analyze oil samples retrieved directly from engines. Results are shown on the analyzer's display and contain alarms so users can make informed decisions immediately.

This set of tools all originated through a joint effort with the U.S. military aimed at developing a condition-based oil change program. The tools are used in the field to reduce costs and improve reliability. Now, maintenance professionals have the power to make decisions in the field, which makes condition-based oil changes both affordable and practical.

Portable Oil Condition Monitoring Combinations

The set of portable oil condition monitoring tools developed by Spectro Scientific includes an infrared (IR) spectrometer, a temperature-controlled kinematic viscometer, and a portable fuel dilution meter. This triple combination paints a complete picture of in-service oil condition, including oil degradation, coolant contamination, water contamination, fuel contamination, and viscosity. All three tools are battery powered and use less than 1 milliliter of oil combined. The in-service oil parameters for diesel, gasoline, biodiesel, propane, biogas, and natural gas engines that can be tested using the combination kits are: oxidation, nitration, sulfation, anti-wear additive, total base number, water, glycol contamination, soot, fuel dilution, and viscosity.

The FluidScan Q1000 is a handheld IR spectrometer (Figure 1). It measures oil absorbance spectrum in the mid-IR range (2.5 mm–12 mm). Instead of using Fourier transform infrared spectroscopy technology, which was more widely used in oil analysis laboratories, diffraction grating-based optics with detectors is used for better portability and durability. Chemometric calibration is applied on the raw IR spectrum to obtain oil condition information, such as oxidation, nitration, sulfation, anti-wear additive, total base number, water, glycol contamination, and soot. The technology was recently granted an ASTM D7889 standard method.

FluidScan is widely used in laboratories as a titration alternative, in fleet management for mining trucks, in marine vessels, in power generation plants, and in industrial plants for oil condition-based predictive maintenance. The patented flip top cell uses three drops of oil, takes one minute, and does not require any

1. FluidScan Q1000: The handheld infrared spectrometer measures oil absorbance.
Courtesy: Spectro Scientific



chemicals or solvents to clean. The tool also has an onboard database with asset information and preset alarm limits utilizing a traffic light system (Figure 2). As a result, maintenance engineers can make immediate decisions right after the measurement.

2. Danger! Results are flagged using a color-coded system that alerts users to out-of-specification conditions.
Courtesy: Spectro Scientific

Measure Asset » Results	
Asset: Truck1 (111)	
Apolloil EX DH-1 10W-40	
0 hours	
14 Jun 2013 09:38:28	
AW Additive	73 %
Sample Has Bubbles!	
Glycol	0.1 %
Nitration	6.7 abs/mm
Oxidation	0.0 abs/mm
Soot	0.26 %wt
Sulfation	0.0 abs/mm
TBN	0.0 mgKOH/l
Visc-40	0 cSt
<<	Detail Discard Save

Chapter 17: Oil Analysis for Shipboard Maintenance

Naval Sealift Ship Puts Accuracy to the Test

The United States Naval Ship (USNS) Watson is one of the large, medium-speed, roll-on, roll-off ships that have significantly expanded the nation's sealift capacity. The Watson has a considerable amount of machinery, including main engines, generators and cranes, that need oil tested on a regular basis to detect potential problems and eliminate the possibility of a catastrophic failure. Operated by Ocean Shipholdings Inc. under contract to the Military Sealift Command, the Watson previously used oil test kits but found them time-consuming. Its engineers questioned the kits' accuracy, too.

Figure 14-1: USNS Watson under way



Three years ago, the ship switched to handheld lubricant condition monitors, and one year ago added a portable viscometer to its oil testing arsenal.

"The portable instruments help us get more done in less time while providing results that are more accurate and reliable than single-test kits," said Ocean Shipholdings' William Maus, chief engineer of the USNS Watson. "When I show them to engineers from other ships they ask, 'when can we get ours?'"

Major Military Mover

Military Sealift Command operates approximately 110 non-combatant, civilian-crewed ships that replenish

U.S. Navy ships, strategically pre-position combat cargo at sea around the world and move military cargo and supplies used by deployed U.S. forces and coalition partners.

The Watson can carry a variety of military equipment in support of Army and Marine Corps operations. The Watson and other ships of its class were the major transporters of military equipment during Operations Enduring Freedom and Iraqi Freedom and during the military operations in Afghanistan and Iraq that began after the September 2001 terrorist attacks.

The Watson is 950 feet long, has a beam of 106 feet, a fully loaded displacement of 62,644 tons, and a service speed of 24 knots. It has a cargo carrying capacity of more than 380,000 square feet — equivalent to almost eight football fields. There are two gas turbine engines, each with an output of 32,000 brake horsepower (bhp), driving two shafts with 24-foot controllable pitch propellers at 95 rpm at full power. The ship's diesel generators are capable of producing 12,500 KW of electrical power. The ship has many hydraulically powered cranes, cargo doors and ramps.

Figure 14-2: U.S. Army tracked vehicles are loaded aboard the roll-on, roll-off ship USNS Watson



Oil: Critical to Mission

"Our engineering department consists of 11 people, which is not a lot for a 950-foot ship," Maus said. "We are responsible for millions of dollars of machinery which could at any moment become critical to our national defense. As in all major Navy ships, oil analysis plays a critical role on the Watson by alerting us to problems that have the potential to damage a vital system." The information provided by oil analysis enables Maus and his team to efficiently allocate scarce resources and to plan maintenance based on actual need as opposed to simple intervals of time.

In the past, Watson engineers used test kits for oil analysis. Engineers collected oil samples, brought them back to the control room, and mixed them with the chemicals in the test kit. It was necessary to perform tests in the control room in order to maintain a stable environment for the test chemicals and for the test equipment. The chemicals used in the testing process are classified as hazardous, which poses problems for shipping the chemicals and disposal of the used reagents.

Normally, it takes about five minutes to collect a sample, five minutes to bring it back to the control room, and five minutes to perform each of the five tests required for generator oil, for a total of 35 minutes. Maus was concerned about accuracy and repeatability because the tests were dependent on using the right amount of both oil and chemicals, and on the reliability of the kit's base equipment. He was also concerned about the need to work with hazardous chemicals.

Alternatives were found with the Spectro FluidScan Q1000 handheld lubricant condition monitor, and the SpectroVisc Q3000 portable viscometer. The Q1000 performs 87 tests and provides 174 results in about two-thirds the time it took to conduct 138 tests yielding 138 results with the chemical test kit. But Watson's operators wanted to be sure these test results were accurate and reliable, not just fast.

Head-to-Head Comparison

The USNS Watson was one of two Military Sealift Command ships asked to perform a head-to-head comparison of the traditional one-test-at-a-time kits versus portable instruments that operate on the same principles as laboratory instruments. To evaluate the performance of the kits versus portable instruments, Watson personnel were asked to perform a specified number of tests in triplicate — once on a test kit, one on a portable instrument, and also by sending a sample to a lab on shore.

The FluidScan Q1000 is a rugged, handheld infrared spectrometer that measures a range of key oil condition parameters in both synthetic and petroleum-based lubricants and fluids. It can determine lubricant contamination, degradation and cross-contamination at the point of use by measuring key oil condition

Figure 14-3: Oil viscosity results are visible in the Q3000 display



parameters. FluidScan can readily determine total acid number (TAN), total base number (TBN), oxidation, nitration, sulfation, additive depletion, incorrect lubricant, water, glycol, soot, glycerine and FAME (fatty acid methyl esters) in biodiesels.

The SpectroVisc Q3000 was designed to determine kinematic viscosity in the field, for applications when immediate results are required to determine the health of critical equipment. This portable, battery-operated instrument has a touch-screen interface and is designed to be easy to use. Requiring no solvents, no density checks and no thermometer, the SpectroVisc Q3000 measures each sample at a constant temperature, for consistent accuracy without pre-test measurements.

Figure 14-4: Testing air compressor oil aboard the USNS Watson, using the Q3000



Figure 14-5: The Q1000 handheld



Hours Saved

“The first thing we noticed was that Spectro’s instruments greatly simplify the process of measuring oil conditions,” Maus said. “The instruments are light and don’t require any chemicals or extra steps, so you can carry them to the machinery and perform the analysis on site.” Additional benefits he noted were a major reduction of the quantity of oil required for testing, and no generation of hazmat that requires disposal. “The FluidScan Q1000 measures the full range of oil condition parameters in about five minutes, the same time that is required to measure a single parameter with a test kit,” Maus said. He calculated that it takes five minutes to take a sample of oil from a diesel generator, five minutes to measure critical parameters on the oil condition analyzer at the generator, and five minutes to measure the viscosity, for a total of 15 minutes. That’s less than half the time required with test kits.

“When we are sitting in port and not operating a lot of equipment, we save about 10 hours per week,” Maus said. “When we are under way the savings are approximately 15 hours per week. The responsibility for oil analysis is assigned to a licensed engineer with many other responsibilities, so these time savings make his job much easier and allow more efforts to be focused on maintenance and repair throughout the ship.”

Proving Accuracy

A key part of the head-to-head assessment was comparing the accuracy of the portable instruments to the test kits. Testing by an independent laboratory showed that the portable instruments were consistently accurate over the full range of measurement parameters. The accuracy of the test kits, on the other hand, varied from good to poor depending on the specific test and the care taken by the person running the test.

For example, problems were identified in the measurement of TBN with the test kits. This measurement is used on diesel engine oil to measure additives used to neutralize acids produced as a byproduct of combustion. The test kit measurement is based on and requires entry of the original TBN in the oil. Ship’s engineers entered this value based on the specifications provided by the oil manufacturer; however, the accuracy of these specifications was in doubt. The FluidScan Q1000, on the other hand, accurately measures TBN without being dependent on manufacturers’ specs. The portable instruments also provide many additional measurements at no additional cost or time.

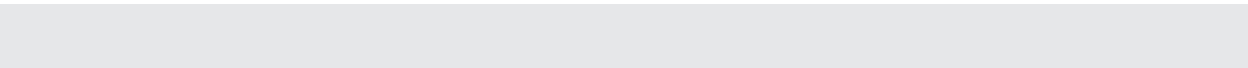
Maus added that another benefit of using portable instruments is that their higher accuracy increases the confidence in their results and leads to greater predictive maintenance efforts. “The accuracy of the portable

instruments gives us confidence to base our predictive maintenance program on their results. We can track exactly what’s going on and identify problems in plenty of time to take corrective action. At the same time, if the results look good we can extend the service life of the oils to save time and money.” In addition to the advantages this equipment offers, the portable instruments are actually less expensive than the test kits previously used, which required replenishment of chemicals and periodic recalibration of the base unit.

“At the end of the trial period, our guys did not want to give up the Spectro instruments and go back to the test kits,” Maus said. “The Navy agreed that the Watson could continue to use the Spectro kits while they continued their evaluations. We are hopeful they will decide to switch the entire fleet over to the new portable instruments. The end result will be increased accuracy and more measurements, which in turn will provide higher mission readiness, lower maintenance expenses, and time savings that can be applied to other shipboard projects. Our future goal is to integrate the results from the new instruments into the ship’s computer based maintenance program so measurement results will automatically be available to shore-side personnel within 24 hours after samples are tested.”









About Spectro Scientific

Spectro Scientific, an ISO 9001:2008 company, specializes in analytical tools and software for machine condition monitoring. The company is one of the largest suppliers of oil, fuel and fluid analysis instruments to industry and the military worldwide. Industry clients include petrochemical, mining, marine and power generation companies as well as commercial testing laboratories. Spectro Scientific's extensive product offerings include spectrometers for wear metal analysis, lubricant degradation and contamination analyzers, particle analysis instruments and complete turnkey systems for oil or fuel analysis laboratories, all managed by its SpectroTrack software platform.

Spectro Scientific, Inc.
One Executive Drive, Suite 101
Chelmsford, MA 01824 USA

Spectro Scientific (Beijing)
Rm. 211, Building 1#,
Hongdalide Industrial Park
Hongda South Road, BDA
Beijing, PRC. 100176