Spectro Scientific
Confidence in knowing

HB0001

Spectro Scientific
Confidence in knowing

Predictive Equipment Maintenance

Oil Analysis Handbook

Preface

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

The initial objective was to compile a comprehensive reference guide of common in-service oil analysis techniques to help readers understand and choose the right technique and instrumentation for their needs. As we started working on the project, it quickly became apparent that the field of in-service oil analysis is so broad and complicated, that it is difficult to cover all the topics and techniques in a short handbook format. So instead, we shifted our focus on the techniques easily deployed by reliability professionals in the field and the products supported by our extensive sales and service network worldwide. This work is not possible without the time and effort from the contributing authors, including Patrick Henning, Daniel Walsh, Robert Yurko, Thomas Barraclough, Aifeng Shi and Yuegang Zhao from Spectro Scientific and Ray Garvey from Emerson Process Management. Special thanks to Sandra Schiller for the excellent management of the project and to Kelly Fisher for the great editorial work and formatting of this handbook.

The handbook starts with an introduction to in-service oil analysis and oil sampling techniques. It is followed by overviews of some key oil analysis techniques and applications. It concludes with several case studies that demonstrate significant return on investment examples after implementing in-service oil analysis solutions. I hope you enjoy reading the handbook and welcome any feedback and critique to make this handbook better in the future.

Yuegang Zhao

Contents

Preface

Chapter 1: Oil Analysis Introduction

Chapter 2: Oil Sampling Best Practices

Chapter 3: RDE Spectroscopy

Chapter 4: LaserNet Fines® Q200 Particle Analyzer

Chapter 5: Portable Viscometer

Chapter 6: Infrared Spectroscopy for Oil Analysis

Chapter 7: FluidScan® Portable Infrared Spectrometer

Chapter 8: Water Measurement Using the IR Technique

Chapter 9: TAN-TBN Measurement Using IR Technique

Chapter 10: Fuel Dilution Meter Overview

Chapter 11: Lubricant Mix Up - How to Catch it with Oil Analysis

Chapter 12: Route-Based Oil Analysis

Chapter 13: Railway Case Study

Chapter 14: Marine Case Study

Chapter 15: Industrial Plant Case Study

Chapter 16: Mining Case Study

Appendix 1. A Detailed Laboratory Report

1: Oil Analysis Introduction

The Importance of Lubricant and Fluid Analysis in Predictive Maintenance

Machine condition monitoring or predictive maintenance is the practice of assessing a machine's condition by periodically gathering data on key machine-health indicators to determine when to schedule maintenance.

One of the keys to keeping machinery operating at optimal performance involves monitoring and analyzing lubricant oils for characteristics such as contamination, chemical content and viscosity. Billions of dollars are spent annually replacing machinery components that have worn out due to the inability of the lubricants to perform the required task. Knowing how to interpret changing lubricant properties can increase both the uptime and the life of your mission critical capital equipment. The existence or amount of debris and particles from wearing parts, erosion and contamination provide insights about the issues affecting performance and reliability.

Lubricant, fuel and other key fluid analyses provide critical early warning information indicative of machine failure. Analyzing and trending the data means you can schedule maintenance before a critical failure occurs. The result – higher equipment availability and productivity, lower maintenance costs, lower Total Cost of Ownership (TCO), fewer outages, optimal equipment performance and a greener operation.

Immediate Benefit of In-service Oil Analysis

Lubricants are the life blood of oil wetted machinery. As an important element of predictive maintenance technologies, in-service oil analysis can provide trace information about machine wear condition, lubricant contamination as well as lubricant condition (Figure 1-1). Reliability engineers and maintenance professionals can make maintenance decisions based on the oil analysis results. The immediate benefits of in-service oil analysis include avoiding oil mix up, contamination control, condition based maintenance and failure analysis.

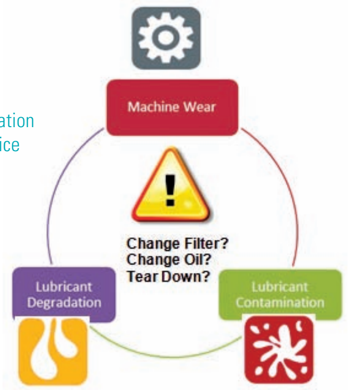


Figure 1-1: Information provided by in-service oil analysis

Avoid Oil Mix Up

The old saying of "Oil is oil" or "when in doubt, put engine oil in," really highlights the under-education of lubricating oils and the importance of optimized lubrication. Oil mix up is one of the most common lubrication problems contributing to machinery failure. Putting the right lubricating oil in the equipment is one of the simplest tasks to improve equipment reliability. Checking the viscosity, brand and grade of incoming new oil, and checking any contamination of alien fluids help reduce the chances of oil mix up and keeps the machine operating.

Contamination Control

Solid contamination (sand and dirt) accelerates the generation of abrasive wear. Liquid contamination such as moisture in oil accelerates machine corrosion. Fuel or coolant dilution in engine oil will decrease the viscosity therefore generating more adhesive wear (rubbing wear). It is critical to keep the lubricating oil clean and dry all the time. This requires that you set cleanliness limits and continue monitoring the contamination during the machine operation.

Oil Condition Based Maintenance

A well balanced oil analysis program can monitor machine wear condition, oil contamination and oil degradation at the same time (Figure 1-1). Key parameters are continuously tested and trending of those parameters is monitored. If a change of rate is accelerated or if a parameter exceeds an alarm limit, reliability engineers are alerted and maintenance actions may be required to resolve the potential problems.

Failure Analysis

A comprehensive oil analysis suite may include tests such as Ferrography, or SEM/EDX which are both time consuming and expensive. However, these tests provide detailed and definitive information about machinery wear, such as what the wear particles are made of, where they come from, and how severe they are. Such information provides reliability professionals with information on a past or imminent failure.

Common In-service Oil Analysis Techniques

Table 1-1 shows typical oil analysis parameters and common analytical techniques to monitor machine wear, contamination and degradation.

Because different types of mechanical components tend to have various oil related issues, different oil analysis techniques might be applied. For example, reciprocal engines tend to generate fine wear particles. Coolant leak, soot buildup and fuel dilution are common problems in lubricants. On the other hand, rotating machinery such as gearboxes tend to generate large wear particles. Acidity increase and moisture contamination are among common parameters monitoring lubricant condition to prevent corrosion. In almost in all cases, monitoring and maintaining lubricant viscosity within specification is critical to ensure mechanical components are well lubricated. Table 1-2 shows typical oil analysis parameters and how they relate to problems by equipment types.

Category	Key Analysis	Analytical Techniques
Machine Wear	Fine wear metals	Rotating Disc Electrode (RDE) Spectroscopy*, Inductive Coupling Plasma (ICP) Spectroscopy
	Large wear metal elements	Rotode Filtration Spectroscopy (RFS)*
	Partice count and distribution	Particle count, LaserNet Fine (LNF)*
	Wear particle shape analysis	LNF*, Ferrography, Wear Debris Analysis (WDA)
Contamination	Sand and Dirt	Particle count, LaserNet Fines (LNF)*
	Fuel Dilution	Fuel Sniffer*, Gas Chromatography (GC)
	Water/Moisture	Infared (IR)*, Karl Fischer Titration (KF)
	Glycol/Coolant	IR*
	Soot	Soot meter, IR*
	Alien Fluid	IR*
Degradation	Oxidation, Nitration, Sulfation	IR*
	Viscosity	Viscosity*
	Acid number (AN), or Base Number (BN)	Titration, IR*

Table 1-1: Key oil analysis parameters and corresponding analytical techniques. Tests with asterisks (*) are highlighted in later chapters of this handbook

Category	Diesel Engines	Gear Systems	Hydraulic Systems
Elemental	Wear contaminants, additives Viscosity - contamination from soot or fuel	Gear boxes generate all sorts of wear, but the levels can sometimes get confusing	Will often validate or clarify particle count: added value for additive and contamination
Particle Count			Quantitative, somewhat holistic
Ferrography		Tracks the large iron-laden particles for Analytical Ferrography trigger	
Fourier Transform Infrared Spectroscopy (FTIR)	Oxidation, nitration combustion by products, glycol contamination	Oxidation, base stock integrity	Oxidation, base stock integrity
Viscosity	Contamination from soot or fuel	Always useful and worthwhile	Always useful and worthwhile
TAN		Contamination or degradation	Contamination or degradation
TBN	Reserve alkalinity, detergency		
Water	Mostly to identify an invalid sample	To validate the sample	Any deductable amount is probably abnormal or critical need particle count validity check
Fuel Dilution	Excessive idling or mechanical issue, such as a nozzle dribbling or leaking injector seal		
Fuel Soot	Combustion cycle indicator, Air fuel ratio		

Table 1-2: Different oil analysis parameters by machine applications.

Common Oil Analysis Practices

There are several ways to perform in-service oil analysis. The most common ones are outsourcing to an off-site laboratory, using an on-site laboratory, or performing route-based oil analysis using portable tools.

Off-site Laboratories (Commercial Labs, Third Party Labs, Central Industrial Labs)

Outsourcing oil analysis to an off-site oil laboratory is probably the oldest and most common approach in the industry. Every year millions of oil samples are analyzed by laboratories worldwide. A typical process flow involves a user collecting oil samples from equipment and shipping them to a lab, lab technicians performing requested oil analysis tests, and an analyst reviewing the data and providing recommendations. The report is then sent to the management team for review and if needed, maintenance actions are performed taking into account the recommendation and maintenance schedule (Figure 1-2).

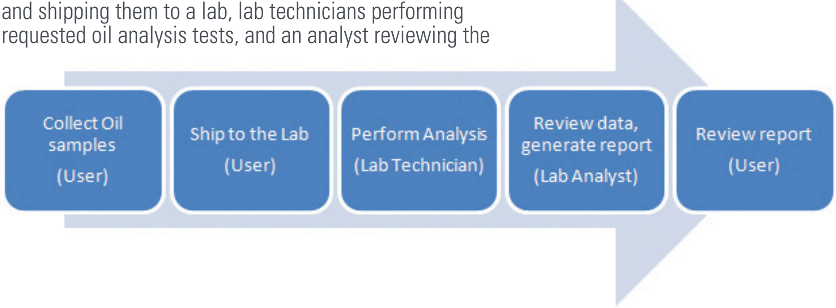
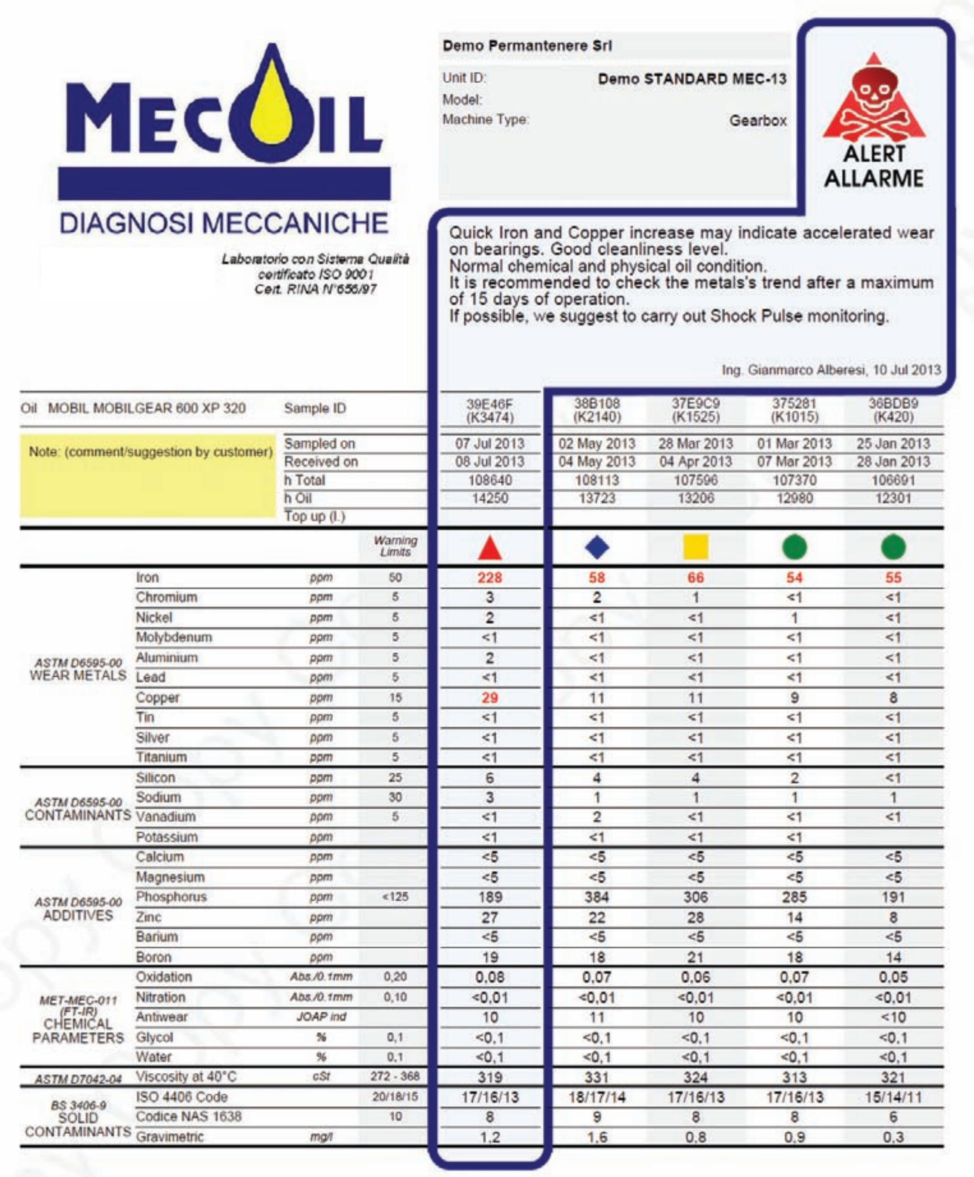


Figure 1-2: Off-site oil analysis flow chart

The benefit of this approach is that an off-site commercial laboratory will have a complete set of oil analysis instruments and experienced technicians to run the tests and an experienced analyst to review the data. The ramp up time is faster and the up-front capital investment is relatively low.

Figure 1-3 shows a typical laboratory report with all the measurement data regarding oil condition and recommendations from the lab.



MECOIL

DIAGNOSI MECCANICHE

Laboratorio con Sistema Qualità
certificato ISO 9001
Cert. RINA N°656/97

Demo Permantenere Srl

Unit ID: **Demo STANDARD MEC-13**
Model:
Machine Type: **Gearbox**

ALERT
ALLARME

Quick Iron and Copper increase may indicate accelerated wear on bearings. Good cleanliness level.
Normal chemical and physical oil condition.
It is recommended to check the metals's trend after a maximum of 15 days of operation.
If possible, we suggest to carry out Shock Pulse monitoring.

Ing. Gianmarco Alberesi, 10 Jul 2013

Oil MOBIL MOBILGEAR 600 XP 320	Sample ID			39E46F (K3474)	38B108 (K2140)	37E9C9 (K1525)	375281 (K1015)	36BDB9 (K420)
Note: (comment/suggestion by customer)	Sampled on			07 Jul 2013	02 May 2013	28 Mar 2013	01 Mar 2013	25 Jan 2013
	Received on			08 Jul 2013	04 May 2013	04 Apr 2013	07 Mar 2013	28 Jan 2013
	h Total			108640	108113	107596	107370	106691
	h Oil			14250	13723	13206	12980	12301
	Top up (l.)							
			Warning Limits	▲	◆	■	●	●
ASTM D6595-00 WEAR METALS	Iron	*ppm*	50	228	58	66	54	55
	Chromium	*ppm*	5	3	2	1	<1	<1
	Nickel	*ppm*	5	2	<1	<1	1	<1
	Molybdenum	*ppm*	5	<1	<1	<1	<1	<1
	Aluminium	*ppm*	5	2	<1	<1	<1	<1
	Lead	*ppm*	5	<1	<1	<1	<1	<1
	Copper	*ppm*	15	29	11	11	9	8
	Tin	*ppm*	5	<1	<1	<1	<1	<1
	Silver	*ppm*	5	<1	<1	<1	<1	<1
	Titanium	*ppm*	5	<1	<1	<1	<1	<1
ASTM D6595-00 CONTAMINANTS	Silicon	*ppm*	25	6	4	4	2	<1
	Sodium	*ppm*	30	3	1	1	1	1
	Vanadium	*ppm*	5	<1	2	<1	<1	<1
	Potassium	*ppm*		<1	<1	<1	<1	
ASTM D6595-00 ADDITIVES	Calcium	*ppm*		<5	<5	<5	<5	<5
	Magnesium	*ppm*		<5	<5	<5	<5	<5
	Phosphorus	*ppm*	<125	189	384	306	285	191
	Zinc	*ppm*		27	22	28	14	8
	Barium	*ppm*		<5	<5	<5	<5	<5
	Boron	*ppm*		19	18	21	18	14
MET-MEC-011 (FT-IR) CHEMICAL PARAMETERS	Oxidation	*Abs./0.1mm*	0,20	0,08	0,07	0,06	0,07	0,05
	Nitration	*Abs./0.1mm*	0,10	<0,01	<0,01	<0,01	<0,01	<0,01
	Antiwear	*JOAP ind*		10	11	10	10	<10
	Glycol	*%*	0,1	<0,1	<0,1	<0,1	<0,1	<0,1
	Water	*%*	0,1	<0,1	<0,1	<0,1	<0,1	<0,1
ASTM D7042-04	Viscosity at 40°C	*cSt*	272 - 368	319	331	324	313	321
BS 3406-9 SOLID CONTAMINANTS	ISO 4406 Code		20/18/15	17/16/13	18/17/14	17/16/13	17/16/13	15/14/11
	Codice NAS 1638		10	8	9	8	8	6
	Gravimetric	*mg/l*		1,2	1,6	0,8	0,9	0,3

Figure 1-3: A typical oil analysis report from an oil lab. Notice the diagnostics and recommendations on the top of the report from the analyst based on the oil type, machine type and oil analysis results (Courtesy of Mecoil).
]

On-site Laboratories

Modern predictive maintenance practices require real-time data and immediate feedback so reliability engineers can make maintenance decisions on the spot. Advances in oil analysis instrumentation technologies also make it possible for reliability engineers to perform on-site oil analysis without years of training or deep knowledge of oil chemistry and testing. There are two common approaches to develop an on-site oil lab.

Depending on the industries and applications one can either set up a Trivector™ Minilab or an Industrial Tribology Lab (ITL).

Trivector™ Minilab

A Trivector™ Minilab (Figure 1-4) was designed to meet the needs of industrial reliability engineers to monitor oil conditions of rotating equipment such as gearboxes, compressors, and turbines. It is the most cost effective approach for a comprehensive on-site oil analysis test lab. It provides oil parameters indicative of machine wear, contamination and degradation (chemistry) and plots the information on an innovative and straightforward Trivector™ chart. In addition, the oil condition information can then be integrated into other predictive maintenance techniques such as vibration and thermal imaging which provide a comprehensive overview of machine condition. The comprehensive Minilab includes tests such as dielectric (oil degradation), water contamination, ferrous particles, particle count, viscosity and wear debris analysis. It is easy to use and the entire test suite can be completed in less than 10 minutes.

Figure 1-4: Trivector™ Minilab and Trivector™ plot

Industrial Tribology Lab (ITL)

A total Industrial Tribology Lab (ITL) contains 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 1-6). However, the instruments in an ITL may be different from a high volume commercial lab. It is designed for small footprint (suitable for on-site or in a trailer), no sample preparation (suitable for reliability professionals), low consumable cost, low waste stream, and ease of use. In fact, the original ITL concept was designed for military applications where the tests are performed by soldiers in the field and where ease of use is a must. The ITL is configurable based on the types of tests required. It is commonly used for remote mining sites, railway repair depot, shipboard, or the central lab in an industrial plant.

The core instrumentation of an ITL includes a RDE Optical Emission Spectrometer (OES), a direct imaging particle analyzer based on LaserNet Fines technology, a temperature controlled viscometer, and an infrared spectrometer and information management software (see Figure 1-5).

The RDE-OES (Spectroil Q100) provides an elemental breakdown of fine and dissolved wear metal particle concentration in parts per million (ppm). It uses 2ml of oil sample and only takes 30 seconds to analyze up to 32 elements at once and complies to ASTM D6595. In addition to in-service oil analysis, calibration programs including coolant, light fuel, heavy fuel, and wash down water can be added to the instruments.

Grease can also be analyzed on the instrument with little sample preparation for trending purposes. Compared to ICP, which is commonly seen in high volume commercial labs, the advantage of the RDE-OES is flexibility, ease of use and no sample preparation. The results from RDE-OES correlate with those from ICP really well.

The LaserNet Fines (LNF) particle analyzer uses direct imaging technique with a high speed CCD camera to capture particles in oil and with Neural Network the software automatically classifies particles larger than 20 m into cutting wear, sliding wear and fatigue wear based on shape analysis. The whole process takes less than 5 minutes comparing 30 to 60 minutes for ferrography. It complies with ASTM D7596. Bitmap images of wear particles from the camera display on the final report.

The viscometer (SpectroVisc Q300 or Q310) is a temperature controlled high speed semiautomatic viscometer compliant with ASTM D7279. If the number of samples per day is low, a manual viscometer (SpectroVisc Q3050) can be used. The advantage of the manual viscometer is that no solvent is needed for after test cleaning.

The infrared spectrometer (FluidScan) is based on diffraction grating optics instead of the FTIR technique. It complies with ASTM D7889 which provides a dedicated oil analysis spectrometer based on diffraction grating and more repeatable and reproducible oil chemistry information than the FTIR method. It also includes a large on-board oil library with accurate TAN, TBN and dissolved water information without the needs of titrators. Data from all the pieces of instruments can be exported to the SpectroTrack information management software which can be installed on site as well as hosted in the cloud.

With additional requirements, a fuel sniffer or/and a soot meter (ASTM D7686) can be added to the ITL configuration, commonly used for maintaining a fleet of diesel engines, such as railway and mining applications. Ferrography (ASTM D7684, ASTM D7690) can be added in the ITL configuration as well for advanced failure analysis.

The ITL configuration is compact, with low oil consumption (low waste stream), low solvent consumption (low recycle cost), and low consumable cost (low cost of ownership). No annual calibration is required on instruments, only standardization and a regular performance check is needed.

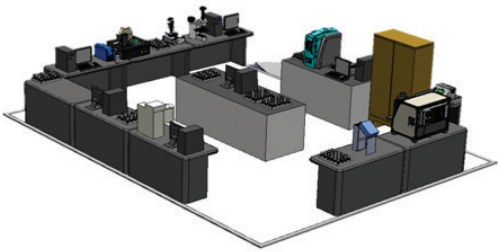


Figure 1-5: Example of an ITL configuration

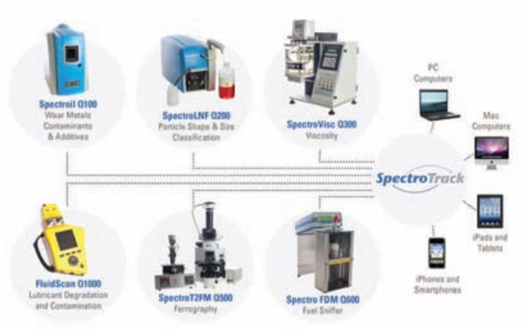


Figure 1-6: Minimum ITL configuration

Mobile Oil Analysis Tools and Route Based Oil Analysis

The advancement of technology makes it possible to have portable, battery powered oil analysis tools with similar analytical power of those instruments used in the labs. Increasingly, more new products are introduced to enable reliability and service engineers to make oil analysis decisions right at the equipment site instead of collecting oil in bottles. This is similar to the ways vibration and thermal imaging is done in an industrial plant. Now reliability service engineers can combine the information from oil analysis and vibration analysis in real-time, which empowers them to make much more informed decisions about machine maintenance. Figure 1-7 shows two examples of portable oil analysis tools. The product to the left is a FluidScan and Viscometer Combination Kit for route-based oil condition monitoring. The product to the right is the Q5800 Expeditionary Fluid Analysis System including a particle counter, a patch maker for microscopy, an XRF for wear particle elemental analysis, a direct IR spectrometer (FluidScan) for oil chemistry and a temperature controlled kinematic viscometer.

All the technologies in the Q5800 are packaged in a suitcase and powered by a lithium ion battery for up to 4 hours of continuous testing. It is very powerful to have the analytical capability of an oil analysis lab in a suitcase so that reliability service professionals can carry it around to critical equipment for just in time oil analysis. In addition, communication options such as cellular modem, wifi or Bluetooth can be employed to transfer the data to a LIMS such as SpectroTrack, so customers may review the data as soon as it is complete.

Figure 1-7: Viscometer Combination Kit (left) and Q5800 Expeditionary Fluid Analysis System

Conclusion

In summary, in-service oil analysis provides critical information about machine condition and oil condition. There are many different approaches to implement an oil analysis program, depending on the application and maintenance objectives. Modern technologies have enabled reliability professionals to use new tools for the highest level of efficiency and effective maintenance.

2: Oil Sampling Best Practices

Introduction

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. Getting a representative sample is critical to effective oil analysis, whether analysis occurs on-site or at an off-site laboratory. Sampling techniques vary by machine and application; manual sampling concepts are detailed in ASTM D4057. Equipment makers also have recommended procedures to ensure samples are taken at the right place and the right time.

Non-Uniform Distribution of Particles

Because 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 slow moving sections, 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 distributed evenly throughout the oil piping system. Consequently, in order to capture a representative sample, the sampling location must be chosen carefully.

Life of a Particle

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 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 are now reduced in size by various chemical and physical processes. The larger particles are relatively new and are better evidence of the current condition of the wearing surfaces.

It is the larger particles in an oil sample that represent the current wear condition; the small particles are primarily the residue of history.

Time to Reach Particle Equilibrium After an Oil Change

When a machine is started after an oil change, coarse particle concentration will increase and the removal rate by settling and filtration also increases, until such time when the generation rate and removal rate are in equilibrium. The time to reach equilibrium varies from machine-to-machine and even within a machine because of the following variables:

- Very fine filters cause equilibrium to be reached quickly; systems without filters reach equilibrium very slowly.
- The gas turbine of a military aircraft pumps the oil system volume in seconds and reaches equilibrium in minutes; the oil volume of a steam turbine may be pumped in many minutes and the time to reach coarse particle equilibrium is measured in hours or even days.
- If the flow is turbulent throughout a system, the settling rate reduces so it takes longer to reach a higher equilibrium population.

Where to Take a Sample

A representative oil sample should be taken at the same location using the same method each time, as the following table indicates:

Table 2-1: Sampling Points Per Equipment Type

Equipment	Sampling Point
Engine Crankcases	• Through dipstick holder • Sample valve on crankcase sump wall • Return line before filter
Hydraulic Systems	• Sampling Valve on return line before filter • Reservoir- about mid point, away from reservoir walls
Compressors	• Crankcase: about mid point away from crankcase walls • Return line after oil separator
Gearboxes	• Sump: mid point. Avoid sump floor sludge and sidewall deposits
Turbine Systems	• Reservoir- mid point-keep away from sidewalls and baffle plates • Main bearing return line • Secondary points- After bypass filtration system

How to Sample

Oil sampling is normally done with either a sampling thief pump (non-pressurized systems) or directly from a sampling valve (pressurized systems). Sampling valves are recommended as a way to standardize sampling on both pressurized and non-pressurized equipment.

Sampling from Pipes

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

Sampling from Tanks

- Take sample from the tank center and well clear of bottom or sides.
- 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 stand off to prevent bottom sampling, as well as clasps to hold the tygon tubing.

Labeling Samples

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

Sampling Dos and Don’ts

- DO sample when the machine is operating, or within 30 minutes of operation.
- DO always sample from same point.
- DO immediately label the sample to prevent mix up.
- DO drain off dead leg oil/water/debris before sampling.
- DO NOT sample immediately after an 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 to consider when 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 (is it wet, dry?)
- 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 sample interval is appropriate for most important industrial machinery, whereas reciprocating engines tend to be sampled at a more frequent interval based on time on 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 on-site 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 guide is designed as an aid to establishing a proper interval.

Equipment Type	Normal Use Sampling Frequency (Hours)	Normal Use Sampling Frequency (Calendar)	Occasional Use Sampling Frequency (Calendar)
Industrial and Marine			
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	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

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

Equipment Type	Normal Use Sampling Frequency (Hours)
Aviation	
Reciprocating Engines	50 hours
Gas Turbines	100 hours
Gearboxes	100 hours
Hydraulics	100 hours

Table 2-2: Equipment Type Per Sampling Frequency

Sample Bottles

The use of clean bottles and caps, together with proper labels and convenient shipping containers, help to make an oil analysis program efficient and effective. Most oil analysis samples for off-site testing use a 38 mm wide mouth HDPE or polystyrene (clear) bottle; the mouth chosen because it is a defacto standard for vacuum sampling pumps. Industrial equipment being sampled off-site requires a 120 ml bottle (traditionally because of the high volumes needed for optical particle count), whereas reciprocating engines use a 60 ml bottle. Certified, super clean bottles may be purchased for contamination control programs.

Sampling Accessories for Portable Equipment

Proper sampling valves, sampling pumps, tubing and other accessories make oil sampling easier and more repeatable–an important objective.

Training

A person experienced in oil sampling should walk through a process with the trainee, advising him/her on each machine to be sampled. Follow up with documentation on the method of sampling and the necessary tools.

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, meaning oil analysis results help you save equipment downtime and provide overall cost savings.

3: RDE Spectroscopy

Introduction

The basis of modern oil analysis 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 else an oil lab may measure, a multi-elemental analysis is the core of in-service oil analysis. This paper gives an overview of Rotating Disc Electrode Elemental Spectroscopy and its use for in-service oil analysis applications.

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. Today, spectrometric oil analysis is widely applicable to any 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. The spectrometer analyzes the sample for trace levels of metals worn from moving parts as well as contamination and additive element levels. The resulting data, when compared to previous analyses and allowable limits, may indicate a sound mechanism 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.

Spectrometric oil analysis works because fine particles are generated by relative motion of metallic parts in an oil-wetted system. The lubricating oil may be thought of as a diagnostic medium because the oil carries with it the particles generated by the wear contact. Abnormal wear modes such as corrosion, abrasion, severe wear, spalling, etc., cause an increase in the concentration of wear metals in the oil. Contaminants are detected and lubricant mix ups or badly degraded lubricants are identified by the concentration of additive elements. Multi-element analysis, coupled with knowledge of the materials of construction, often allows identification of a specific component in distress. Table 3-1 shows typical metal elements can be analyzed by spectroscopy and their sources.

So how does a spectrometer work?

Table 3-1: Typical Source of Elements Analyzed by Spectroscopy in Oil

Metal	Engine, Transmission, Gears	Hydraulic Fluid	Coolants
Aluminum Al	Pistons or Crankcases or Reiprocating Engines, Housings, Bearing Surfaces, Pumps, Thrust Washers	Pumps, Thrust Washers, Radiator Tanks	Coolant Elbows, Piping, Thermostat, Spacer Plates
Barium Ba	Synthetic Oil Additive Synthertic Fluid	Additive	Not Applicable
Calcium Ca	Detergent Dispersant Additive, Water Containment, Airborne Contamination	Detergent Dispersant additive, Water Contaminant, Airborne Contamination	Hard Water
Chromium Cr	Pistons, Cylinder Liners, Exhaust Valves, Coolant Leak from Cr Corrosion Inhibitor	Shaft, Stainless Steel Alloys	Corrosion Inhibitor
Copper Cu	Either brass of bronze allot detected in conjunction with zince for brass allloys and tin for brass alloys. Bearings, Bushing, Thrust Plates, Oil Coolers, Oil Additive	Bushings, Thrust Plates, Oil Coolers	Radiator, Oil Cooler, Heater Core
Iron Fe	Most common or wear metals. Cylinder Liners, Walve guides, Rocker arms, Bearing, Crankshaft, Camshaft, Wrist Pins, Housing	Cylinders, Gears, Rods	Liners, Water Pump, Cylinder Block, Cylinder Head

Table 3-1: Typical Source of Elements Analyzed by Spectroscopy in Oil (continued)

Metal	Engine, Transmission, Gears	Hydraulic Fluid	Coolants
Lead Pb	Bearing Metal, Bushings, Seals, Solder, Gease, Leaded Gasoline	Bushings	Solder, Oil Cooler, Heater Core
Magnesium Mg	Housing on Aircraft and Marine Systems, Oil Additive	Additive, Housing	Cast Alloys
Molybdenum	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, Airbirne Contaminant	Coolant Leak, Airbirne Contaminant	pH Buffer
Silicon Si	Airborne Dusts, Seals, Coolant Leak, Additive	Airborne Dusts, Seals, Coolant Leak, Additive	Anit-foarming and Anti-corrosion Inhibitor
Silver Ag	Bearing Cages (silver plating), Wrist Pin bushing on EMD Engines, Piping with Silver Solder Joints from Oil Coolers	Silver Solder Joints from Lub 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

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. When subjected to the addition of energy, each element emits light of specific wavelengths or colors. Since no two elements have the same pattern of spectral lines, the elements can be differentiated. 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. The light has a specific frequency or wavelength determined by the energy of the electron in transition. Since many transitions of different energy are possible for complicated atoms which have many electrons, light of many different wavelengths is emitted. If this light is dispersed by using a dispersing element such as a prism, a line spectrum will result. These spectral lines are unique to the atomic structure of only one element. For the hydrogen atom with atomic number 1, the spectrum is fairly simple (Figure 3-1). On the other hand, the spectrum of iron with atomic number 26 is much more complicated with many emission lines in the visible spectrum corresponding to the many possible electronic transitions that may occur (Figure 3-2). 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 possible choices is chosen to determine the concentration of a certain element. This line will be chosen for its intensity and freedom from spectral line interference of other elements.
To accomplish this, an optical system is required.

Rotating Disc Electrode Optical Emission Spectroscopy (RDEOES)

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, these components are:

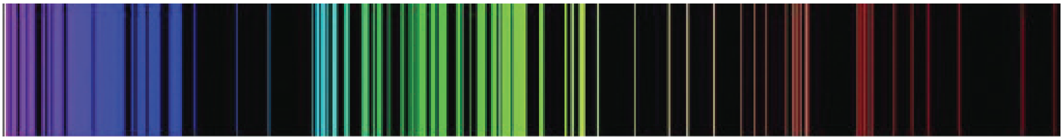
1. Excitation Source - introduces energy to the sample.
2. Optical System - separates and resolves the resulting emission from that excitation into its component wavelengths.
3. Readout System - detects and measures the light that has been separated into its component wavelengths by the optical system 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 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 hard to excite elements emitted 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. This requires about 2 or 3 ml of sample depending on the exact cap being used. A fresh disc and a newly sharpened rod are required for each sample to eliminate sample carryover. This method is called RDE optical emission spectroscopy (OES), or combining the two, RDEOES. Alternatively, one often sees it referred to as RDE¬AES, for rotating disc electrode atomic emission spectroscopy.

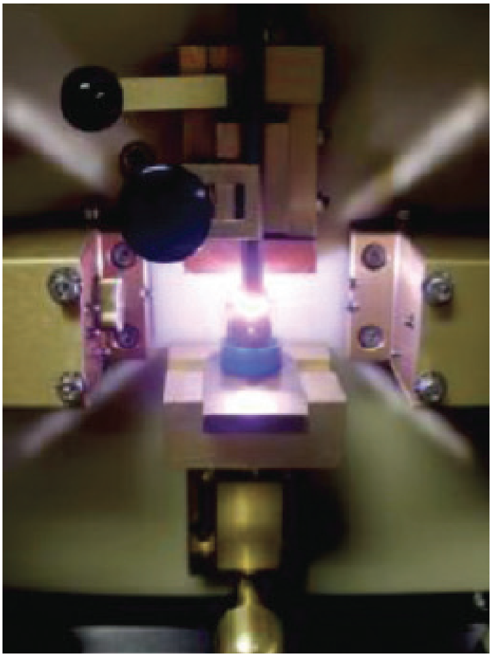
Figure 3-1: Emission Spectrum of Hydrogen

Figure 3-2: Emission Spectrum of Iron



The light coming from the plasma is separated by the optical system, in a spectrometer, into the discrete wavelengths of which it is comprised. An optical device called a diffraction grating is used to separate the discreet 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.

Figure 3-3: RDE Spectrometer Sample Stand Showing Oil Sample being "Burned"

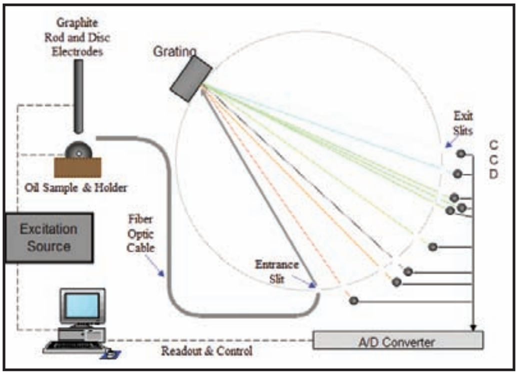


The following schematic shows the major components of an oil analysis spectrometer using a polychromator optic based on the Rowland Circle concept. Light from the excitation process (burn) exits the fiber optic cable and passes through the entrance slit and is concentrated on the diffraction grating 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 (diffract) this light into its component wavelengths. The spectral lines can be photographed or electronically quantified by photomultiplier tubes (PMTs) historically, or today, charged couple device arrays. An important consideration when designing a spectrometer is the region of the spectrum where the wavelengths of interest occur. Many elements emit light in the visible region of the spectrum. However, there are elements that emit mainly in the Far Ultra Violet (FUV) region of the spectrum. This is significant because FUV radiation does not transmit well through air; rather, it is mostly absorbed. For the optic to see FUV spectral lines, it is necessary for the optical system to be mounted in a vacuum chamber or otherwise filled with gas transparent to FUV light, so the emitted light can reach the grating, be diffracted, and then be detected at the focal curve.

Thus, a sealed chamber and a vacuum pump or gas supply system become part of the system.

The readout system of a spectrometer is typically controlled by an industrial grade processor and software. A clocking circuit and amplifier periodically reads the charge on a Photo Multiplier Tube or CCD chip and converts it from an analog to 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. Concentration is usually expressed in parts per million (ppm). This information is displayed on a video screen or can be printed out on a printer. Once the analysis is completed and the results recorded, the system is ready for the next analysis.

Figure 3-4: Schematic of Rotating Disc Electrode Optical Emission



The analysis results may be left on the screen, stored on the hard disk, or can be sent to an external computer.

Older generations of spectrometers are heavy and bulky, such as the one below, a Baird FAS-2C spectrometer from the 1970's, many of which were deployed at various military bases throughout the world and were also installed on aircraft carriers. This was a rather large instrument with a one meter optic (the distance from the grating to the focal curve) and weighing about 800 lbs (364 kg). With original factory crating it weighed 1400 lbs (636 kg). It was hardly a field mobile instrument!

Figure 3-5: A Direct Reading Oil Analysis Spectrometer from the 1970's

After decades of development and improvement, RDEOES today is much more compact and user friendly. Figure 3-6 shows the Spectroil Q100, which weighs only 163 lbs (74 kg) with a very small footprint while still maintaining the same analytical capability as the bigger systems in previous generations. Today, Spectro Inc.'s Spectroil M and Q100 are widely used in elemental analysis for lubricant, coolant and fuel for mining industry, power plants, as well commercial laboratories serving industrial plants for predictive maintenance and quality control. In addition, different performance enhancement options are available for special applications.

Automation for the RDE spectrometer has always been difficult due to the need to replenish the graphite electrodes after each analysis. The rod electrode, in particular, has been a challenge to handle by robotics, since it not only must be sharpened after each burn but also becomes shorter after each sharpening. The practical solution to RDE spectrometer automation is to use two graphite disc electrodes (Figure 3-7). This eliminates the need to sharpen electrodes and greatly simplifies operation. The automation system consists of two parts: a robot to exchange the consumable disk electrodes and an automatic sample changer. The robot dispenses new graphite electrodes for each analysis and removes the used ones. The need to set the analytical gap size has also been eliminated since the disk electrode shafts form a fixed gap. A robotics arm in the sample changer automatically introduces each of 48 oil samples in succession, at a rate of 80 samples per hour and 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 operating software. Although operation is automatic, it also has the capability to manually sequence through each of the robotics functions. Automation also improves repeatability by eliminating operator variances and maintains cross unit correlation.

Figure 3-6: The Spectroil Q100 RDE Spectrometer

Large Particle Size Analysis Capability

The ability to detect and quantify large wear and contaminant particles has been considered one of the shortcomings of spectrometers. Few will agree as to the actual detrimental impact on a condition monitoring program, but most will agree that the practical particle size limitation of spectrometers are at particles below 5 micrometers for ICP and AAS and at particles below 10 micrometers for RDE [1, 2]. Today, the particle size limitation of RDE spectrometers has been eliminated with simple ancillary systems such as the Rotrode Filter Spectroscopy (RFS) method.

RFS makes use of the fact that the carbon disc electrodes used in RDE spectrometers are porous. A fixture is used to clamp the discs so oil can be drawn through the outer circumference of the discs when a vacuum is applied to the inside of the discs (Figure 3-8). The particles in the oil are captured by the disc. The oil is then washed away with solvent, the disc is allowed to dry, and the particles are left on the disc electrode so they are vaporized and detected when run on the RDE spectrometer. It is a technique whereby the normal analysis of the oil sample serves to provide data on particles dissolved to 10 micrometer in size, and the RFS technique analysis provides data on large particles [3]. A multi-station fixture is used so a number of samples can be filtered at one time. Several commercial laboratories offer RFS to provide a more comprehensive analysis of used oil samples.

Figure 3-7: Robotics for RDE Spectrometer

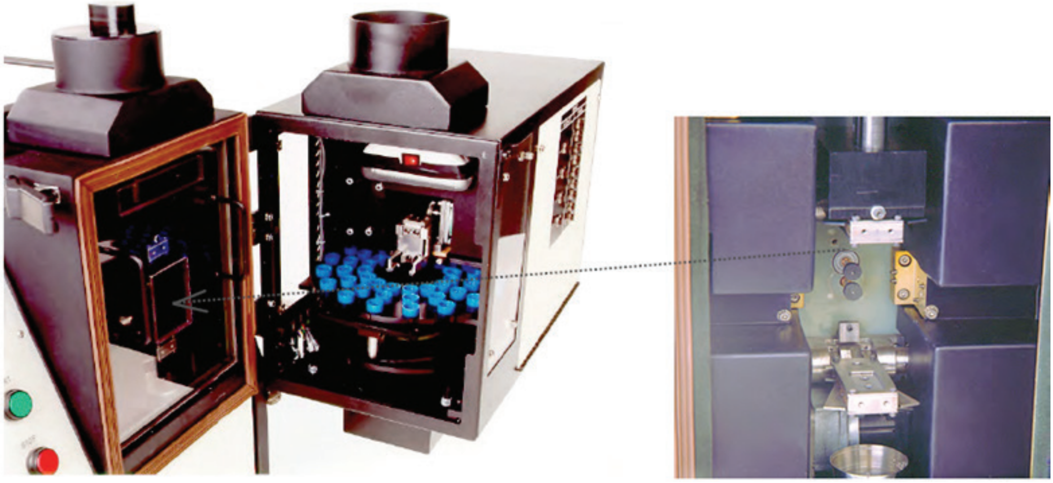
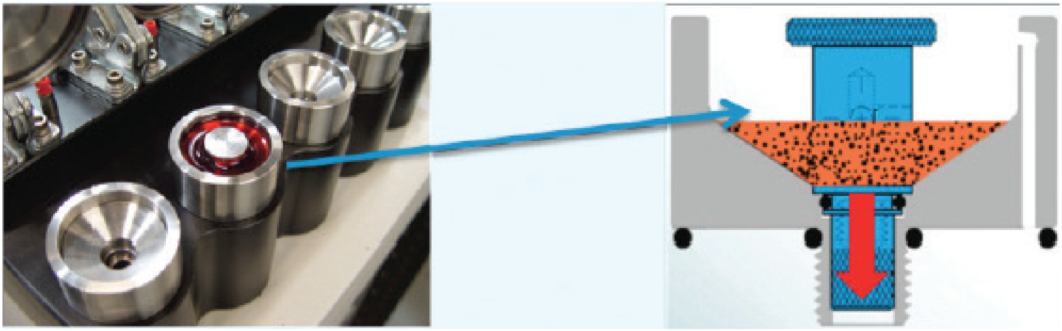


Figure 3-8: Automatic Rotrode Filter Spectroscopy (A-RFS) Fixture



Extended Application Development

Although the RDE spectrometer is still designed primarily for used oil and fuel analysis, several methods and recent enhancements have increased productivity through expanded capabilities. They include the ability to analyze engine coolants, sulfur in oil and fuel, etc. A used coolant analysis program determines both the coolant condition and the presence of any contaminants or debris. The coolant fluid can be used as a diagnostic medium as the coolant carries heat away from the engine parts as well as fine debris from the interior surfaces of the cooling system. Analysis of the wear debris can provide important information about the condition of the internal parts of the cooling system.

Some machine condition monitoring programs have gone beyond used oil analysis and also provide data on the coolant system. The application of coolant analysis; however, has been limited due to additional cost and the time required to analyze the samples. Today, several major commercial oil analysis laboratories have switched to the RDE technique for coolant analysis. This was made possible with minor hardware and software modifications to the RDE spectrometer. It has been shown that the RDE technique correlates well with Inductive Coupling Plasma (ICP) and AAS techniques on new coolants and is more efficient on used coolants that contain particulates [4].

A case where the RDE spectrometer helped a big saving for a customer in one incident can easily demonstrate its value. A serious recurring problem in maintenance procedures is the use of an incorrect lubricant. A condition monitoring program can readily identify such problems through the analysis of the lubricant additive package and lubricant physical property analysis.

Lubricant mix ups often occur when an oil system is “topped off ” to replace the oil that has been lost due to use or leakage. Usually a small amount of incorrect oil in a large closed loop system presents few immediate problems. This is, however, not the case in certain diesel engines as illustrated by this example [5].

The data clearly shows that after the first two samples, an incorrect oil was used to top off the reservoir. The three additive metals, magnesium (Mg), phosphorus (P), and zinc (Zn), appear in the third analysis and increase in the fourth -- a clear indication that the oil formulation has changed. 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 cause by the zinc additive are indicated by the increase in the iron, copper and silver wear metals.

A recommendation based on the analysis was made to drain and flush the system and to observe correct top-off oil requirements. This particular fault would not have manifested itself 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. Table 3-2 is a summary of the last four spectrometric oil analyses for a medium speed diesel engine from a locomotive. Only the data for the most relevant elements are shown.

Table 3-2: Spectrometric Results in ppm for a Medium Speed Diesel (EMD) Locomotive

	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
11-Jun	25	30	10	220	110	123

Conclusion

In summary, RDE Optical Emission Spectroscopy has come a long way and today has been widely used in many industries as a reliable tool to analyze fluid samples for condition monitoring and quality control applications.

4: LaserNet Fines® Q200 – A Comprehensive Solution to Oil Analysis Including Particle Count and Particle Shape Classification

Summary

This paper describes how using the state-of-the-art LaserNet Fines® (LNF) Q200 instrument 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. 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 cases 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.

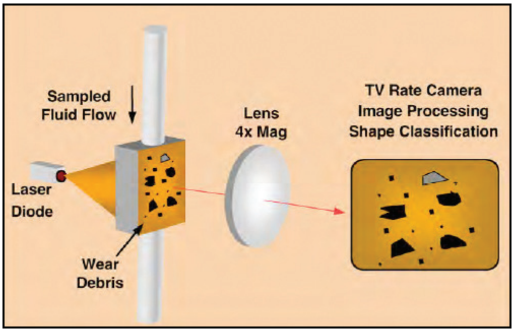
Introduction – LNF Technology

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 20µm, 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.

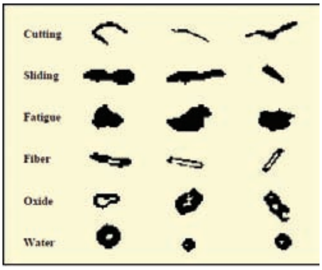


Figure 4-2: Examples of Particle Class Morphology

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 250um were reported using conventional particle counters.
- When interfering soft particles are present in the oil (such as water droplet, 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.

	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

Table 4-2: Consideration Factors

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.

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 below compares Analytical Ferrography with LNF according to various analysis factors.

	Prep Time	Analysis Time	Debris ID	Ferrous/ Nonferrous ID	Free Water	Operator Skill Level	Results
Analytical Ferrogaphy	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

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.

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 15 µm in size. The number of particles greater than 20 µm 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

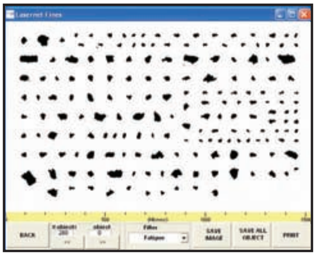


Figure 4-4: Image Map for Engine Test Cell Sample

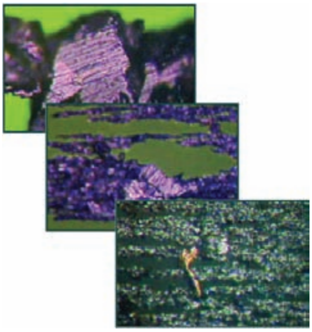


Figure 4-5: Ferrograms Showing Severe Sliding Wear and Copper Particle during Break-in

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 Montoring) 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 5-15µm 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.

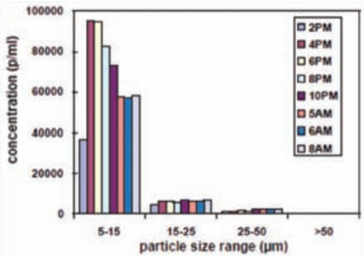


Figure 4-6: Gearbox Total Particle - Concentration Distributions

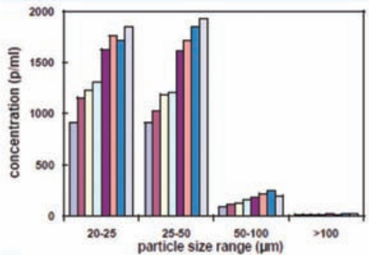


Figure 4-7: Gearbox Fatigue - Particle Concentration Distributions

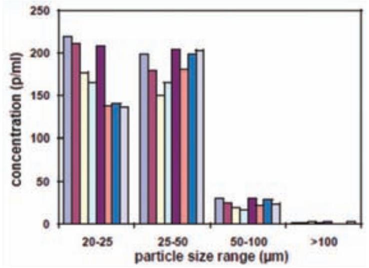


Figure 4-8: Gearbox Severe Sliding Wear - Particle Concentration Dilutions

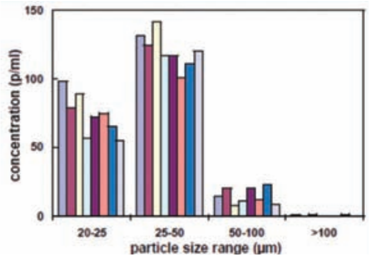


Figure 4-9: Gearbox Severe Cutting Wear - Particle Concentration Dilutions

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 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

5: 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

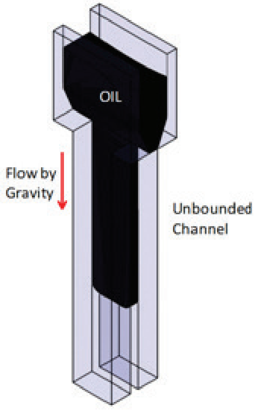
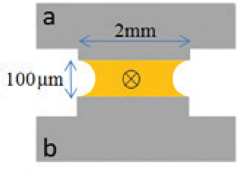


Figure 5-2: Concave Meniscus - Top View



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, 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 dx2 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.

$$\mu \frac{d^2u}{dy^2} + pg = 0 \qquad v = \frac{gd^2}{12U}$$

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 31mN/m. The surface tension induced pressure value at the unbound surface is where R1 is radius of meniscus (half of microchannel depth; 50µm) and R2 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 100mm 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.

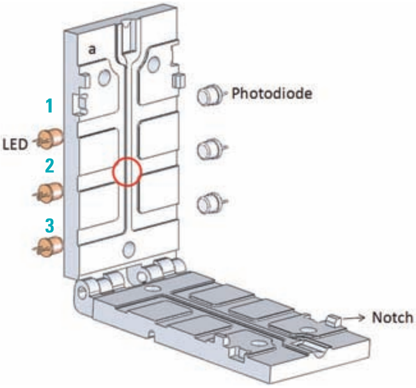
$$\Delta P = \gamma\left(\frac{1}{R_1}+\frac{1}{R_2}\right) \sim 620Pa \qquad \Delta H = \frac{\Delta P}{\rho g} \sim 76mm$$

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%.

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

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.

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

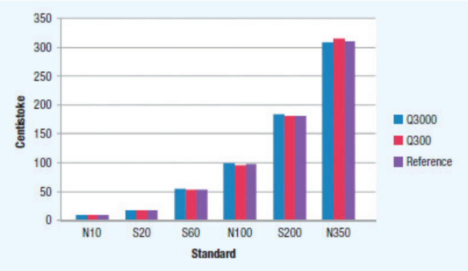


Table 5-1		
Q3000	Ref	% Diff
10.13	10.03	1.00
17.93	18.04	0.61
54.86	54.08	1.44
99.26	97.15	2.17
185.00	180.80	2.32
308.67	310.90	0.72
Portable Viscometer		

Table 5-2		
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		

Table 5-3		
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		

Table 5-4: This table compares used engine oils

Table 5-4			
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

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] Rezazadeh, 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.

6: Infrared Spectroscopy for Oil Analysis

Making the Case for Infrared Spectroscopy in Lubricant Analysis

Why is infrared spectroscopy used in lubricant analysis? To answer the question, we will need to understand what needs to be tested (what comprises lubricant analysis) and what infrared spectroscopy is and can actually deliver.

To begin with the first question: What tests do we generally need to perform to do in-service lubricant analysis? For sure, it depends on the system being monitored, the conditions in which that system may be operating, and what lubricant the system utilizes. However, Table 6-1 shows a generalized list.

Table 6-1 shows that 14 out of 17, or greater than 80%, of in-service lube properties of interest may be monitored using infrared spectroscopy. This clearly demonstrates that infrared spectroscopy can be an important tool for lubricant analysis. Why, then, isn't infrared perhaps the first thing we think of when performing lubricant analysis, if it can provide so many relevant parameters? To answer that, we will need to dig a little deeper.

Next question, then: What is infrared and what can it actually deliver? For example, Acid Number (AN) is by far more common to use titration (such as described in ASTM D664) rather than infrared to accomplish the task. However, infrared spectroscopy is increasingly utilized to monitor AN and BN in all types of systems.

This follows a general trend in the analysis of petroleum products. Infrared, Raman, and other spectroscopic techniques are increasingly becoming accepted as standard tools of choice to perform tests which previously required wet chemical analysis. It is a fact that this trend continues to accelerate.

So what is infrared and why can infrared deliver these properties? The infrared spectroscopy of lubricants relies on a very simple method. You observe how much infrared radiation the lubricant absorbs as a function of 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).

This begs the question, how do we turn these spectra into useful information? This is the key step, and one that has been the key challenge for infrared over the past four decades, when it first began to be utilized

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 6-1: Typical lubricant parameters that are measured and the relevance of infrared spectroscopy per property

for lubricant analysis. The answer is, in general, by applying well-established chemical statistical analysis tools, popularly referred to as “chemometrics”. Such chemometrics analysis relies on creating “calibrations”. Calibrations are very straightforward: You take a range of in-service lubricants which may have very different levels of contamination and degradation, and measure the infrared spectrum of each of these samples. Then, you take these same exact samples and measure the chemical property of interest on a historically standard apparatus, such as a wet chemistry titrator, for each of these samples. What you are left with is a table of sorts — an infrared spectra of individual samples each paired with the property of interest (for example, AN for that sample).

If you have truly taken and measured a representative set of samples from a certain type of lubricant, you then create a calibration. Using chemometrics, you may create a formula which turns the acquired infrared spectrum of a new, unknown sample, into an accurate AN reading for that sample. That is how a calibration works. Now, and going forward, you may take the spectra of any sample of lubricant of this type and immediately infer the AN of that sample by applying that same formula to the spectrum.

This is the true mission of infrared for lubricant analysis: We want to perform this process for as many lubricant products as possible for as many properties as possible. In other words, we want to create a “library” of calibrations which can turn an acquired infrared spectrum into any property you choose, for any lubricant you choose. That’s the goal.

But wait — there are so many types of lubricants in the world. You cannot create a calibration for every property listed in Table 6-1 for every lubricant in the world, can you? Or even every lubricant that you may encounter, right? In fact, this challenge has limited the applicability of infrared analysis over the years and masked its true potential for lubricant analysis. Only recently have we begun to move out of this predicament.

How is this accomplished, or how do you break out of this scenario when there are so many lubricant varieties, making it seemingly necessary to perform endless calibrations and limiting the effectiveness of infrared analysis? We start by doing what we can do – measuring as many lubricant types as we can and looking for commonalities amongst these lubricants. To do this, we make use of statistical classification technologies and good old chemistry: The chemistry will tell us commonalities in the chemical makeup of various market lubricants. We use this to guide the statistical analysis, which merely looks for patterns in the many types of lubricants we at Spectro Scientific have measured.

The result is very exciting to report. Even though there are thousands upon thousands of lubricants in the world, most all (>90%) belong to only a few common families of lubricants. We must be very careful stating that two lubricants belong to a common family. What exactly do we mean by this? What we mean practically is that lubricants in a given family can use the same formula or calibration when turning the acquired infrared spectrum into a lubricant property such as AN. That’s it. So as long as you can identify/verify which family a given lubricant belongs to, and you have done the work to create the

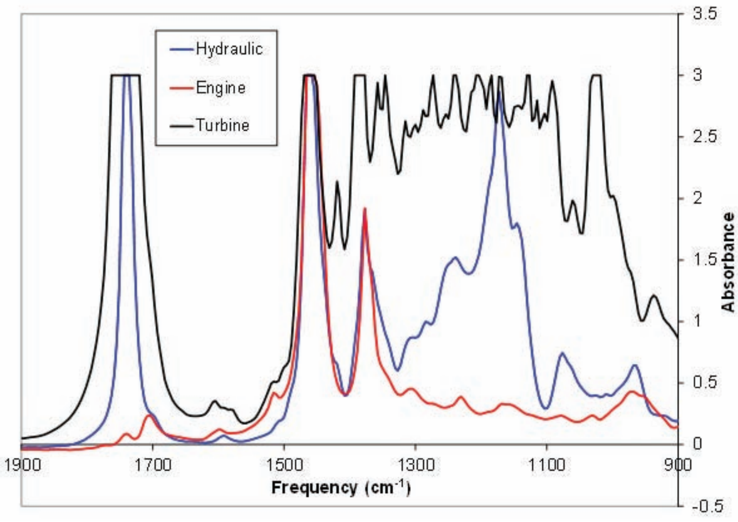


Figure 6-1: Typical lubricant infrared spectra. The interpretation of these spectra allows for monitoring the relevant properties listed in Table 6-1.

calibrations needed for that family, you can take an infrared spectrum of an oil never encountered before and supply quantitative lubricant information.

With this picture in mind, we can create a systematic process for providing these results. Such a process involves three key pieces: (1) creating calibrations for known lubricant families, (2) providing an upfront statistical analysis methodology to identify whether an unknown lubricant belongs to a particular family (done by using well-established statistical classifiers), (3) identifying lubricants which don't belong to a particular identified family, and creating new families for these lubricants. If we keep working at these three steps, we can exponentially expand the applicability of infrared analysis to each and every oil that proves to belong to a given family where relevant classifications exist.

Such an approach to lubricant analysis using infrared spectroscopy (only developed systematically over the past five years) has been proven time and again over its short period of existence that all these properties which infrared analysis "can" provide can be turned into properties that infrared "routinely" provides. This is paving the way for new standards in the industry based on such infrared calibrations.

By realizing that infrared spectroscopy has entered a new era, and need not require "specialized" techniques to provide properties which may be more valuable to the user, we can really begin to see that the case for infrared in lubricant analysis has completely changed in just the past few years.

Because of the tremendous changes in infrared instrumentation, such as the capability to eliminate solvents with wipe-clean cells, the paradigm-shifting reduction in the size of the infrared analyzers (which both enable the use of infrared analysis in places it has never been able to reach before), infrared analysis has never been more useful for lubricant analysis practitioners. And we are only just getting started.

7: FluidScan® Portable Infrared Spectrometer

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 delay in 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.

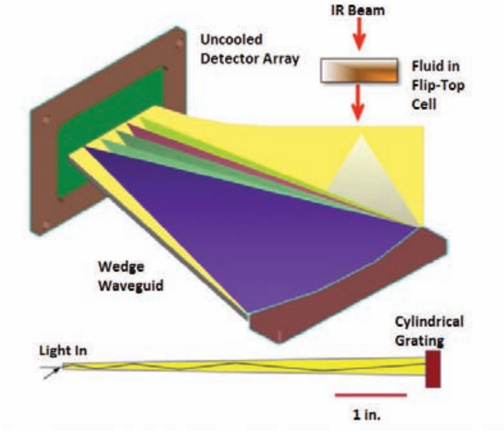
At the core of the FluidScan is a patented, mid-infrared spectrometer with no moving parts. The spectrometer collects the infrared light transmitted through the fluid in the flip top cell into a waveguide as illustrated below.

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

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.

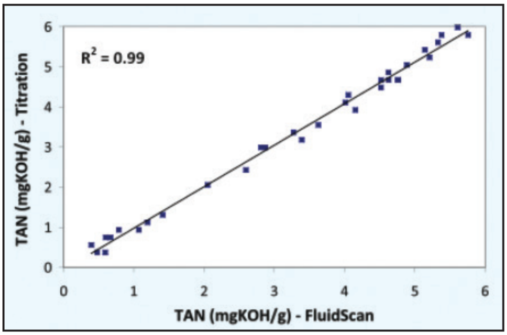
The FluidScan classifies fluids into groups called families based on their chemical makeup, usage and spectral signature. The spectrum of all fluids in each family changes in a similar way with a given amount of degradation or contamination. Family-specific algorithms are assigned that accurately quantify 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 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.

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



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 (Fourier Transform Infrared Spectroscopy) 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 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.

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 (%)

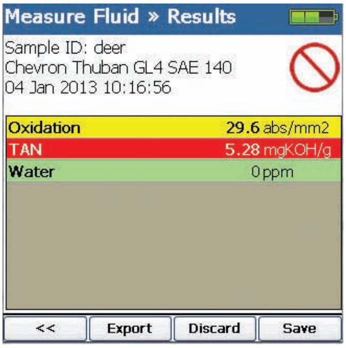
Alarm Limits and Reference Oil Library

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



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

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.

8: Water Measurement Using the IR Technique

Introduction

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.

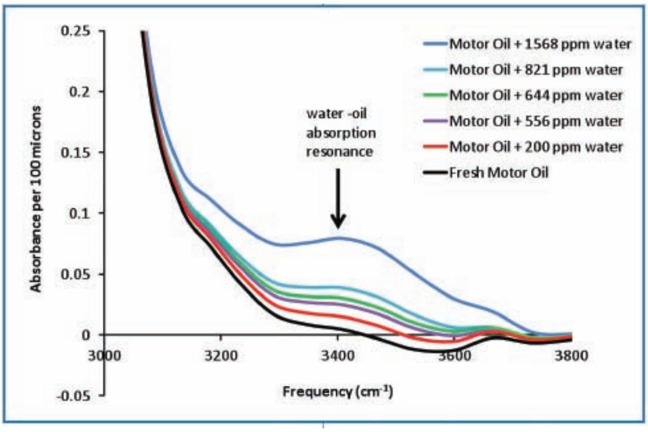
The task of detecting water contamination in oil using a mid-infrared spectrometer such as FluidScan is a natural fit. When water interacts with oil, signature chemical bonds are created between the water and the oil molecules, which have strong mid-infrared resonances. This means the bonded water-oil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

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

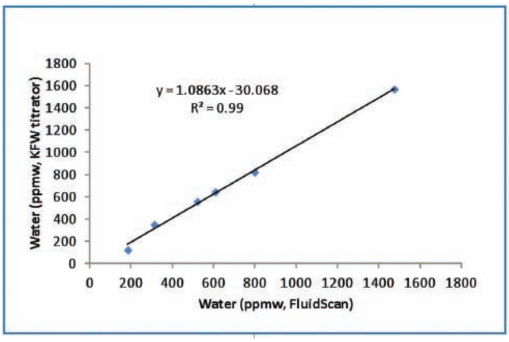
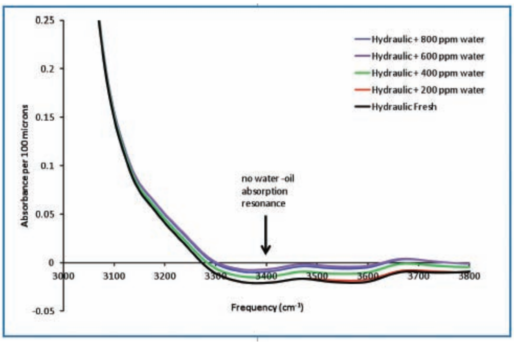


Figure 8-3: Infrared signature of emulsified and free water in oil: The presence of such contaminants is marked by elevated absorption across the infrared with no peak, a signature of the scattering of the infrared light by the water.



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 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 limit due to weathering and the persistent presence of water in the oil. 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.

9: TAN-TBN Measurement Using IR 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

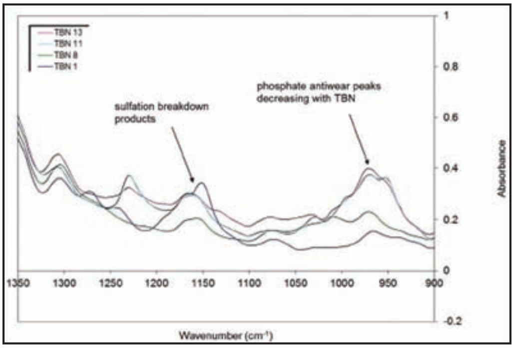
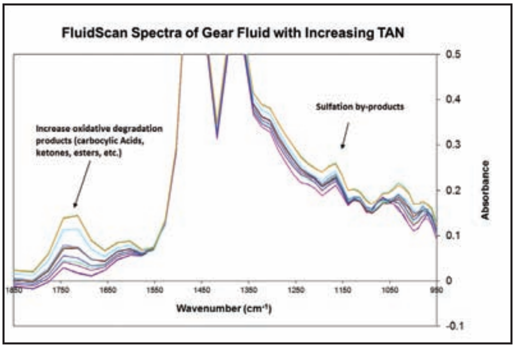


Figure 9-2: Increase of TAN in a gear oil as reflected in the infrared spectrum of the fluid. This data is taken using a Spectro FluidScan



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 (Celcius)	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.

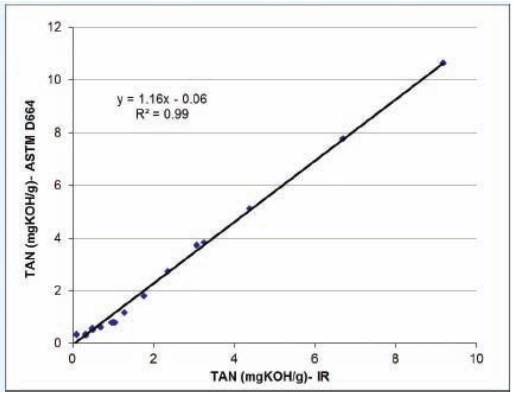
The repeatability and reproducibility of these standard methods for used oils are well-documented. Repeatability is the variability of measurements on the same sample by the same operator using the same measurement equipment. Reproducibility is the comparison of independent tests.

ASTM D664 Reproducibility	ASTM D664 Repeatability
$R = 0.44^{*}x$	$r = 0.117^{*}x$
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.

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



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.

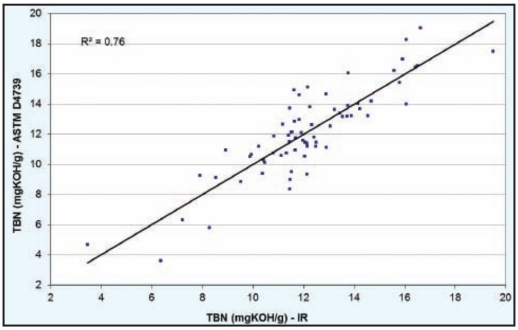
For an in-service sample with a TBN of 10 mg KOH/g, titration results can range from 5.5 to 14.5 mg KOH/g within the specification, with repeatability between 9.6 and 10.4 mg KOH/g.

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
$R = 1.54^{*}x^{0.47}$	$r = 0.22^{*}x\ 0.47$
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

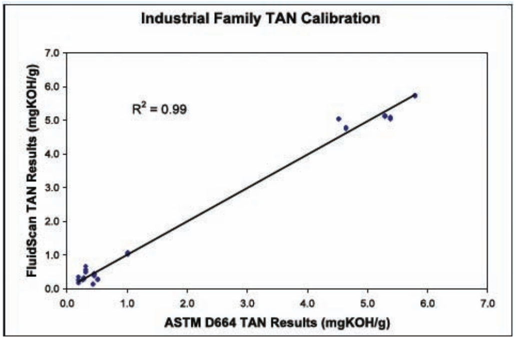


Example of TAN Correlation: Industrial Fluids

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

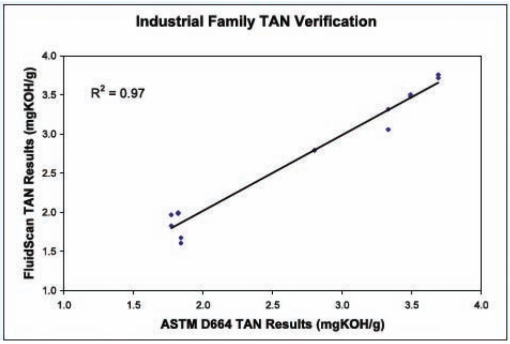


Example of TAN Correlation: Heavy Gear Oils

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.

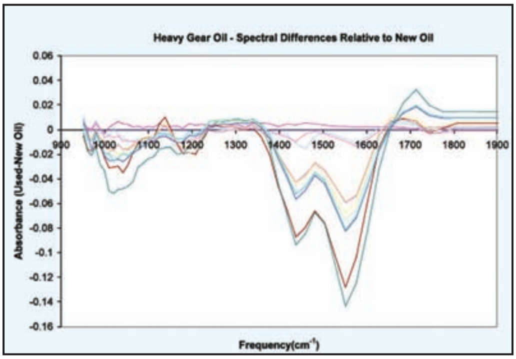


Figure 9-7: Spectral evolution of a heavy gear oil relative to the new fluid shows both increasing and decreasing absorbances at various frequencies as the oil becomes more degraded

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 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.

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.

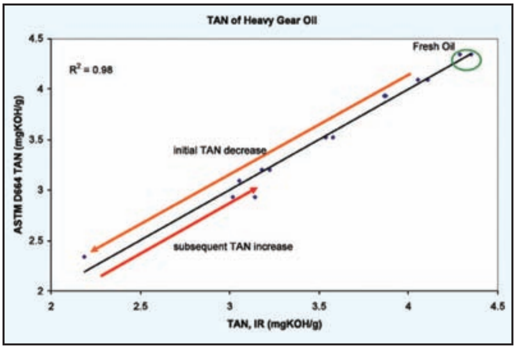
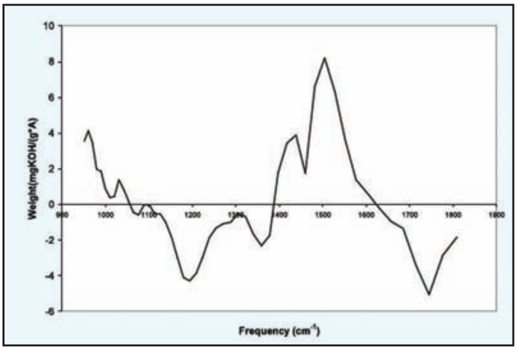


Figure 9-9: Prediction vector for the FluidScan for marine diesel engines



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
Novermber 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 Spectro Scientific'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).

10: Fuel Dilution Meter Overview

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 SpectroFDM Q600 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 (NAVSSES) 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 microsensot. 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.

Using the SpectroFDM Q600 Fuel Sniffer 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. This fuel sniffer requires no chemicals or consumables, making it inexpensive to use as well as fast and accurate.

Today, Fuel sniffer is widely used by maintenance professionals and laboratory technicians around the world supporting automotive, railway, marine, and heavy equipment fleets in operation for engine maintenance.

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.

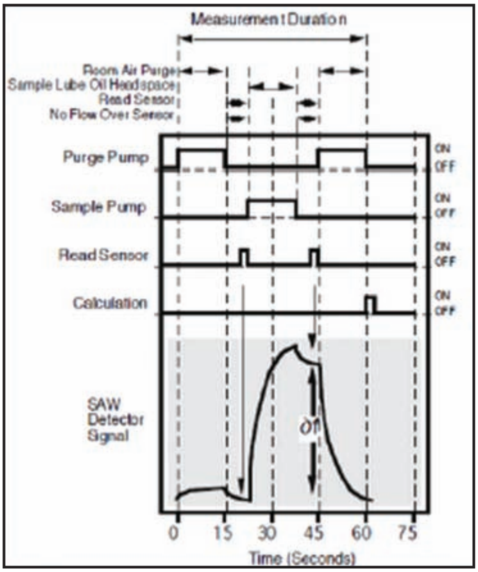
Figure 10-1: SpectroFDM Q600

Principle of Operation

The Fuel Sniffer employs a Surface Acoustic Wave (SAW) Vapor Microsensor to measure the concentration of fuel in used lubricating oil samples by sampling the "headspace" in the sample bottle. The instrument assumes (based on Henry's Law) that the fuel vapor concentration is directly related to the fuel present in the oil sample. A pump inside the instrument draws headspace vapors across the SAW sensor which detects absorbed hydrocarbons by a change in frequency of a surface acoustic wave.

A SAW sensor consists of a piezoelectric substrate that has an interdigital electrode lithographically patterned on its surface. The surface of the SAW sensor has a polymer coating that offers solubility to fuel vapors. The mechanism of detection is a reversible absorption of the fuel component into the polymer. When this device is excited by external RF (Radio Frequency) voltage, a Rayleigh wave (waves perpendicular to the surface) generates on the surface of the device. When fuel contamination comes in contact with the SAW sensor surface it will absorb into the polymer coating.

Figure 10-2: SAW Sensor Signal Response Diagram



This absorption into the polymer causes a mass change, producing a corresponding change in the amplitude and velocity of the surface wave. When used in a self-resonant oscillator circuit, the change in Rayleigh wave velocity resulting from vapor absorption into the polymer coating causes a corresponding change in oscillator frequency. This change in frequency is the basis of the Fuel Sniffer's detection. The absorption is semi-selective based on the properties of the polymer coating and the partition coefficient (solubility of the chemical and polymer) of the chemical of interest. Figure 10-2 shows an example of SAW sensor response in real-time when in operation.

Prior to the Fuel Sniffer, customers relied on GC-MS and Flash Point technologies to detect fuel dilution. **GC-MS** is a method that combines the features of gas-liquid chromatography and mass spectrometry to identify different substances within a test sample. It is known as the "gold standard" for forensic substance identification because it is used to perform a specific test. A specific test positively identifies the actual presence of a particular substance in a given sample. Yes it is powerful, but it is also expensive. The typical cost of a GC-MS is almost 10 times the cost of the Fuel Sniffer to provide good "witness" results on a portable, fast and reliable platform. Also, a highly skilled chemist is required to run the GC-MS instrument to obtain good results.

Flash Point tests can be relatively fast and accurate if an automated flash point tester is used, but it is not as fast or as convenient as the Fuel Sniffer. Flash Point systems do not present percent fuel dilution calculations except when using correlation tables generated by running many samples containing a known percentage of fuel.

In comparison, the Fuel Sniffer offers an improved safety factor over Flash Point methods because it reduces exposure to fire and inhalation hazards from heating fuel samples. In addition, generating correlation tables to obtain the known percentage of fuel is not required with the Fuel Sniffer.

FTIR testing of fuel relies heavily on individual fuel calibrations and careful baselining of the blank oil matrix. The absorbance peak in the spectra regions used to detect fuel is not very sensitive and careful calibration needs to be done by highly trained professionals. Also, different fuels refined from around the world will exhibit different absorbance bands that can sometimes be missed by the calibration peak. The Fuel Sniffer does not suffer from these calibration and matrix problems.

General Operation

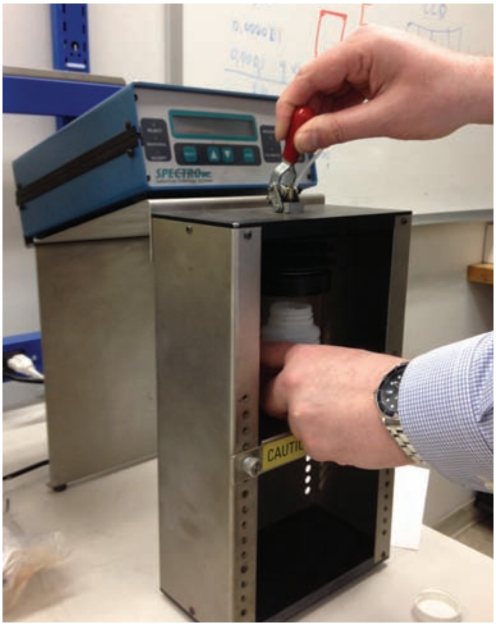
Prior to real measurement, the Fuel Sniffer must be calibrated with a standard concentration containing 5% fuel in a lubricant base similar to the samples to be analyzed. The instrument response to fuel dilution is linear and it makes no provisions to use any other value of calibration standard. If any other level of dilution is used above or below this standard, the instrument will either over or under estimate the actual reading, respectively.

To prepare oil sample bottles:

1. Fill the oil sample bottle approximately full (or about 80 grams).
2. Label each sample bottle.
3. The sensor measures the vapor in the airspace between the sample top surface and the sensor. If the bottle is filled to the top, there will not be enough headspace for an accurate reading. And, if the sample is drawn inside the instrument, the sensor may get damaged.

4. The Fuel Sniffer needs just over 60 seconds to perform each measurement. The next sample can be introduced quickly with the specially designed “clamp and sample” bottle platform, so that 40 or more samples can be measured per hour.

A single-point calibration, using a 5% fuel/oil standard, is as fast to run as a sample, so the Fuel Sniffer is soon ready to make accurate measurements. The accuracy is comparable to that achieved by gas chromatography, but without all the time, expense and inconvenience. The percentage fuel dilution displays on the backlit LCD display and can also be sent to a printer or external computer to make accurate measurements. Users can prepare calibration bearing in mind that the oil and fuel of the calibration must be of the same matrix, or similar matrix, to that being measured. Preparation should be made using gravimetric techniques (by weight) in order to maintain accuracy. A small error may be introduced if the standard is mixed volumetrically (by fluid quantity). Calibration standards can also be purchased through Spectro Scientific.



Case Studies:

The following two case studies further demonstrate the capabilities of the Fuel Sniffer:

Case #1: Repeatability and Accuracy

In the following study, a 5% diesel fuel standard was prepared gravimetrically using an SAE 10W 30 mineral oil as the base. The sample was left open for 20 minutes to remove the light end. The sample was then capped for two minutes prior to being calibrated. Further dilutions were made at 3%, 5%, 8%, and 0% of the 10W 30 mineral oil. These samples were filled to exactly the same position and prepared identically to the 5% calibration standard.

The Fuel Sniffer was calibrated and each sample was run 10 times. The bottles were left capped for two minutes to allow the fuel vapor to rebuild its equilibrium in the bottle's headspace. The top of each sample bottle was wiped each time it was put under the sample stand clamp to try and prevent any contamination which could possibly carry over to the next sample. The clamp was also wiped with a clean paper towel after each sample was run. The results are shown in Table 10-1.

In conclusion, the repeatability and accuracy of the Fuel Sniffer is good and is more than sufficient to detect contamination due to fuel dilution.

Case #2: Fuel Sniffer over GC-MS

One of the most important characteristics of aviation kerosene in military aircraft engines is its ability to act as a coolant for other engine systems, and then dispose of the absorbed heat in the combustion process. With occasional seal integrity failure, it is possible to have fuel dilution enter engine or hydraulic compartments.

Recently, one of the major fighter aircraft suppliers to the USAF was tasked to evaluate a quick COTS (commercially off the shelf) system for detecting fuel in hydraulic oil – a real concern where fuel is used as a coolant.

While flash point testers were considered at first, the OEM supplier found that the effort and training required was quite difficult. Next, Spectro Scientific was contacted to arrange for a demonstration unit to be sent to compare referee laboratory methods, such as GC-MS. Three Air Force bases participated in the study and the local calibration was performed.

The end result was satisfactory to all the parties, especially considering that the Fuel Sniffer system provided good "witness" results compared to a GC-MS system that costs almost 10 times more. A witness test refers to the concept that the fuel sniffer will positively confirm if fuel is present. The GC-MS, with its very low levels of detection was, however, a better option to confirm when no fuel was present (virgin samples). See Table 10-2.

Table 10-1: Repeatability and Accuracy Results

Sample Run #	0% (Blank Oil)	3% Diesel Fuel	5% Diesel Fuel	8% Diesel Fuel
1	0	3	4.8	8.2
2	0	3.2	4.9	8.3
3	0	3	5	8
4	0	3.1	4.8	8
5	0	3.1	4.9	7.9
6	0	3	4.9	8.1
7	0	3	5.1	7.9
8	0.1	3.4	5.1	7.8
9	0.1	3.1	5.3	8.1
10	0	3.2	5.1	7.9
Average %	0.02	3.11	4.99	8.02
Precision %	0.042	0.1287	0.1595	0.1549
EAS %	-------	4.14%	3.20%	1.93%
Absolute Error (Accuracy)	0.02%	0.11%	0.01%	0.02%

Table 10-2: Estimation of JP8 Content in PAO by various techniques

Sample	% JP8 Via Fuel Sniffer	% JP8 Via GCMS
1	0.6	0.16
2	0.3	0.3
3	0.3	0.31
4	0.4	0.35
5	0.3	0.53
6	0.3	0.51
7	0.3	0.63
8	0.3	0.25
9	0.3	0.62
10	0.5	0.62
11	0.6	0.64
12	0.6	0.69
13	0.5	0.65
14	0.5	0.57
15	0.5	0.68
16	0.6	0.66
17	0.7	0.06
18	0.6	0.56
19	0.6	0.53
20	0.7	0.58
21	0.7	0.56
22	0.7	0.48
23	0.6	0.5
24	0.7	0.56

Conclusion

As the case studies illustrate, the SpectroFDM Q600 provides a cost-effective, portable solution to detecting fuel dilution in oil samples. Compared to GC-MS it is substantially less expensive to use for single point calibrations while providing repeatable and accurate results. In addition, it requires no chemicals or consumables and is equally, if not more, fast and precise. It is a perfect tool for reliability engineers' for on-site engine maintenance.

References

[1] Diane Lancaster, "Sniffing for Fuel" Pages 54-55 from "Currents" Magazine, Summer 2004

11: Lubricant Mix Up - How to Catch it with Oil Analysis

Introduction

A recent questionnaire 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.

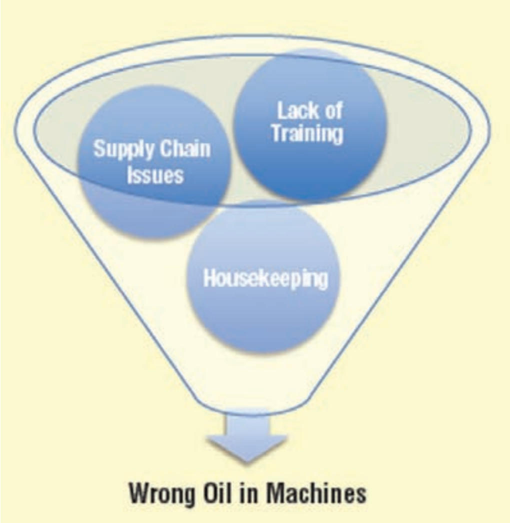
No equipment owner or maintainer plans to put the incorrect oil in a machine. There are three major reasons why it occurs and these reasons, though not exhaustive, are universal across all organizations when this problem is encountered.

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 ispresent.	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 signature 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 smaple
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.

Elemtents	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 scan 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).

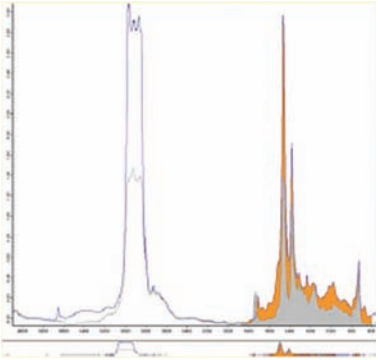


Figure 11-1: Superimposed IR Spectra

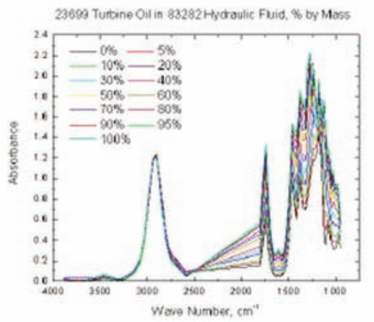


Figure 11-2: Analysis of mix up of oils to determine algorithms, and report out % w/w

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.

12: 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 structrured 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.

Figure 12-1: Route-based Oil Sampling

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 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.

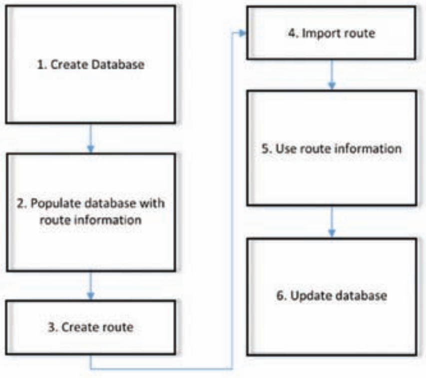
Figure 12-2: Route-based oil analysis observations

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?

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).

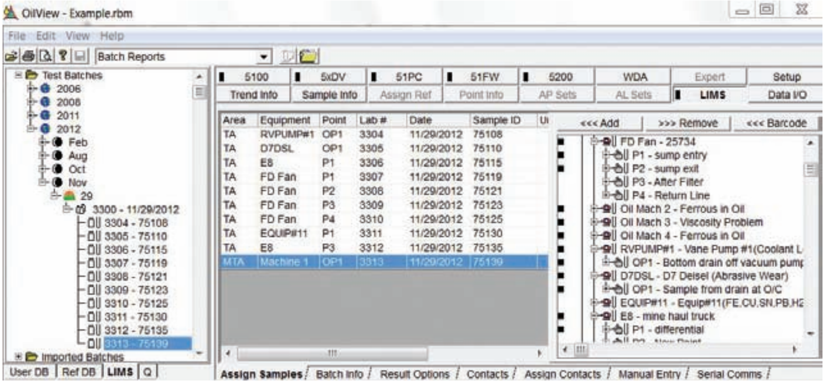
1. Create a structured database in Emerson OilView.
2. Populate the database with route information associated with the oil and equipment in the database.
3. Select a preferred inspection route.
4. Transfer route information into a handheld analyzer.
5. Apply route information in the analyzer during 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.



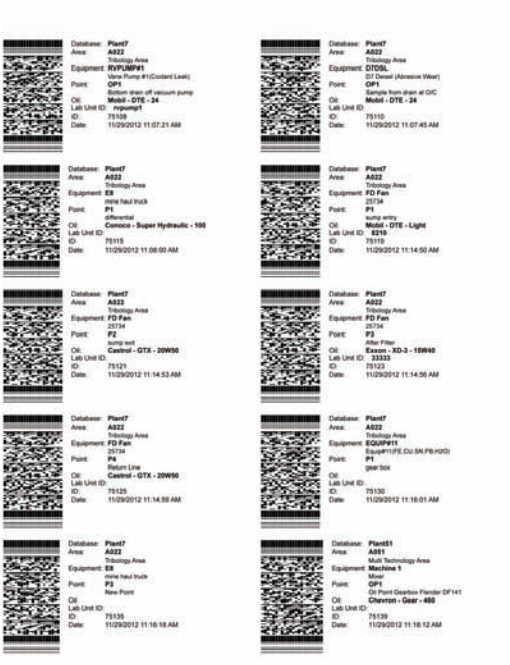
Area	Equipment	Point	Lab #	Date	Sample ID	Ui
TA	RVPUMP#1	OP1	3304	11/29/2012	75108	
TA	D7DSL	OP1	3305	11/29/2012	75110	
TA	E8	P1	3306	11/29/2012	75115	
TA	FD Fan	P1	3307	11/29/2012	75119	
TA	FD Fan	P2	3308	11/29/2012	75121	
TA	FD Fan	P3	3309	11/29/2012	75123	
TA	FD Fan	P4	3310	11/29/2012	75125	
TA	EQUIP#11	P1	3311	11/29/2012	75130	
TA	E8	P3	3312	11/29/2012	75135	
MTA	Machine 1	OP1	3313	11/29/2012	75139	

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.



Database: **Plant7**
Area: **A022**
Tribology Area
Equipment: **RVPUMP#1**
Vane Pump #1(Coolant Leak)
Point: **OP1**
Bottom drain off vacuum pump
Oil: **Mobil - DTE - 24**
Lab Unit ID: **rvpump#1**
ID: 75108
Date: 11/29/2012 11:07:21 AM

Database: **Plant7**
Area: **A022**
Tribology Area
Equipment: **DTDSL**
D7 Diesel (Abrasive Wear)
Point: **OP1**
Sample from drain at O/C
Oil: **Mobil - DTE - 24**
Lab Unit ID:
ID: 75110
Date: 11/29/2012 11:07:45 AM

Database: **Plant7**
Area: **A022**
Tribology Area
Equipment: **E8**
mine haul truck
Point: **P1**
differential
Oil: **Conoco - Super Hydraulic - 100**
Lab Unit ID:
ID: 75115
Date: 11/29/2012 11:08:00 AM

Database: **Plant7**
Area: **A022**
Tribology Area
Equipment: **FD Fan**
25734
Point: **P1**
sump entry
Oil: **Mobil - DTE - Light**
Lab Unit ID: **8210**
ID: 75119
Date: 11/29/2012 11:14:50 AM

Database: **Plant7**
Area: **A022**
Tribology Area
Equipment: **FD Fan**
25734
Point: **P2**
sump exit
Oil: **Castrol - GTX - 20W50**
Lab Unit ID:
ID: 75121
Date: 11/29/2012 11:14:53 AM

Database: **Plant7**
Area: **A022**
Tribology Area
Equipment: **FD Fan**
25734
Point: **P3**
After Filter
Oil: **Exxon - XD-3 - 15W40**
Lab Unit ID: **33333**
ID: 75123
Date: 11/29/2012 11:14:56 AM

Database: **Plant7**
Area: **A022**
Tribology Area
Equipment: **FD Fan**
25734
Point: **P4**
Return Line
Oil: **Castrol - GTX - 20W50**
Lab Unit ID:
ID: 75125
Date: 11/29/2012 11:14:59 AM

Database: **Plant7**
Area: **A022**
Tribology Area
Equipment: **EQUIP#11**
Equip#11(FE,CU,SN,PB,H2O)
Point: **P1**
gear box
Oil:
Lab Unit ID:
ID: 75130
Date: 11/29/2012 11:16:01 AM

Database: **Plant7**
Area: **A022**
Tribology Area
Equipment: **E8**
mine haul truck
Point: **P3**
New Point
Oil:
Lab Unit ID:
ID: 75135
Date: 11/29/2012 11:16:18 AM

Database: **Plant51**
Area: **A051**
Multi Technology Area
Equipment: **Machine 1**
Mixer
Point: **OP1**
Oil Point Gearbox Flender DF141
Oil: **Chevron - Gear - 460**
Lab Unit ID:
ID: 75139
Date: 11/29/2012 11:18:12 AM

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.

13: Railway Case Study

Oil Analysis Helps Keep the Trains Running

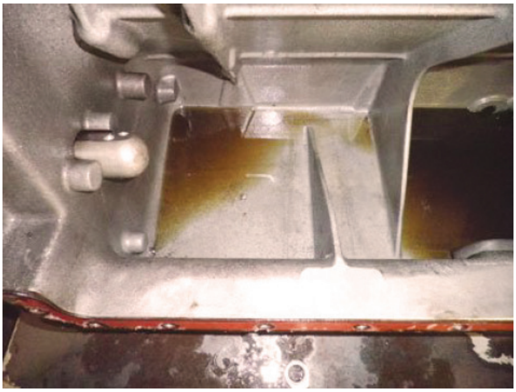
A national railway system got a good look at what state-of-the-art condition monitoring can do for an operation.

Based in Tullow, Ireland, T.E. Laboratories ("the Laboratories"), provides oil, fuel and environmental analysis services for a variety of customers, including Irish Rail, Ireland's national railway system. It regularly uses equipment from Spectro Scientific, including the Spectroil M spectrometer. When the analysis of oil samples from a main locomotive showed a large amount of severe wear particles— indicating that a catastrophic failure was imminent—Irish Rail took the Laboratories' advice and brought the engine in for repair. An overhaul showed chunks of metal visible to the naked eye in the sump and that the bearings were about to fail. In this case, oil analysis saved the railroad both the expense of a more extensive engine overhaul and potential expenses associated with a breakdown. The tally of these avoided costs more than paid for the cost of oil analysis for Irish Rail's entire fleet for a year.

The power of oil analysis

From a custom, 40,000-sq.-ft. facility, T.E. Laboratories provides a machine-care predictive-maintenance system based on condition monitoring through oil analysis. For machine operators, the process is easy: They need spend only a few minutes to obtain an oil sample and ship it to the lab for identification of potential problems. These include abnormal wear in lubricated metallic components, dirty fuel and coolant contamination. Using oil analysis, machinery can be analyzed over a period of time to identify trends that can be used to plan maintenance based on actual need as opposed to simple intervals of time. The Laboratories' services include transformer oil analysis, fuel analysis for microbial contamination and water and soil analysis. Regular customers include trucking, rail and marine shipping fleets; mining, quarrying and construction operators; industrial units; manufacturing plants and heavy-equipment operators.

Figure 13-1: Visible chunks of metal in the sump of the disassembled engine



"As the only oil analysis lab in Ireland, we handle a large number of samples and must deal with them efficiently," says Mark Bowkett, T.E. Laboratories' General Manager. Recent upgrades at the company include implementation of the SpectroTrack laboratory information management system that replaced the company's former "home-grown" system. SpectroTrack offers the built-in ability to interface with the Spectro Scientific, line of viscometers and spectrometers. It can also integrate with instruments from other suppliers for vibration, thermographic and performance data. The database is configured to track asset information relating to service intervals, maintenance actions, locations, status and other issues.

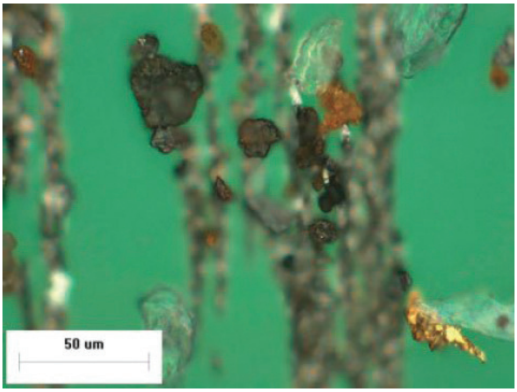
SpectroTrack also provides the Laboratories' customers with security-protected access to their sample results. "The railroad makes extensive use of SpectroTrack Web access to obtain their test reports and to plan maintenance," says Bowkett. "However, we don't wait for customers to notice a problem. When we see something bad, we immediately notify them."

Figure 13-2: A bearing from the engine on the verge of failure

For example, a recent analysis of 18 different wear metals on a liquid-cooled diesel locomotive engine revealed several warning signs. According to the Laboratories' report, the analysis uncovered "the presence of large sliding wear steel particles and some dark metallo-oxides," which indicates "a transition from normal to severe sliding wear and likely due to insufficient lubrication." Additional problems were also noted.

These results "were worrisome, but not necessarily cause for immediate action," says John McGrath, Sales Manager for T.E. Laboratories. But a closer look at the sample was ordered using the SpectroT2FM Q500 analytical ferrography laboratory. This technology uses a bichromatic microscope, video camera and image capture software for the separation and interpretation of wear and contaminant particles in used oils, hydraulic fluids, coolants and fuels. A bichromatic microscope equipped with both reflected (red) and transmitted (green) light sources was used to view and examine ferrograms so that the ferrogram could be illuminated from both above and below the microscope stage. With bichromatic illumination, metal particles that reflect light appeared bright red, while nonmetallic particles appeared green (because light transmits through them).

Figure 13-3: Ferrogram image showing dark metallo-oxides indicative of abnormal wear



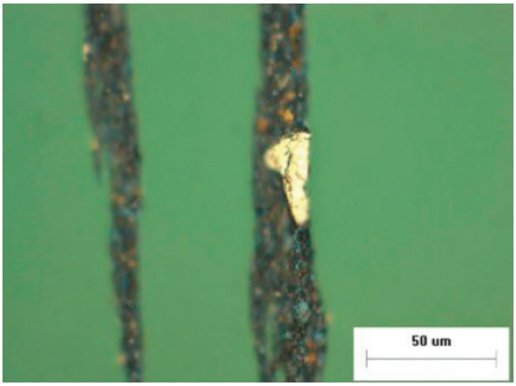
The ferrogram indicated that the problem had, in fact, reached an urgent stage. It showed a moderate to heavy amount of severe copper and white metal wear particles and a moderate amount of dark¬metalloxides. These elements indicate lubricant starvation and abnormal wear. Red oxides were also present, which indicates water ingress. The analysis also showed large abnormal sliding wear particles in excess of 20 microns, copper chunks in excess of 20 microns, non-metallic crystalline particles including silica and rust, low alloy steel fatigue particles and cast iron fatigue chunks from a case-hardened part.

"The particles seen in the ferrogram were quite large and of a critical nature," says McGrath. "The engine was on the verge of destroying itself. It needed to be brought in for an immediate overhaul." When the engine was disassembled, he says, "It was obviously on the verge of a breakdown."

Bowkett believes in the use of cost-efficient oil analysis to prioritize maintenance by avoiding spending money on units that have reached a scheduled milestone, but don't need maintenance. At the same time, he observes, regularly scheduled oil analysis can point out units like the one described above that urgently need maintenance, even though they're not due for service. According to Bowkett, while it may not be possible to estimate the total amount of money saved in the above case by oil analysis, "It's clearly well above the cost of the company's oil-analysis program for its entire fleet."

Figure 13-4: Large copper chunk seen in ferrogram image

Figure 13-5: Cast iron fatigue chunk seen in ferrogram image



14: Marine Case Study

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.

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?'"

Figure 14-1: USNS Watson under way

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 abeam 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.

Figure 14-3: Oil viscosity results are visible in the Q3000 display

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 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.

Figure 14-4: Testing air compressor oil aboard the USNS Watson, using the Q3000

The SpectroVisc Q3000 was designed o 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 14-5: 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."

15: Industrial Plant Case Study

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.

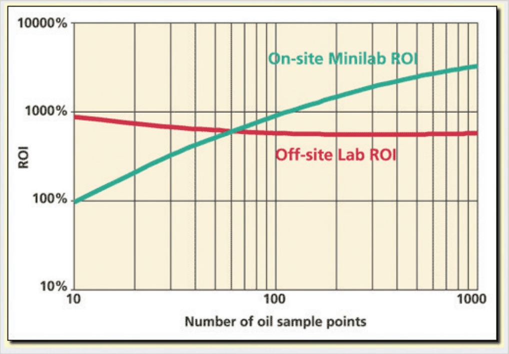
Mike Lawson at the Bowater Paper Mill in Calhoun, TN, would rather test oil at $15 per sample than replace the 35 gallons in a gearbox, the total cost of which would be $480. That includes $140 for the oil, $240 for two mechanics working six hours, $50 to dispose of the used oil, and another $50 to restock.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

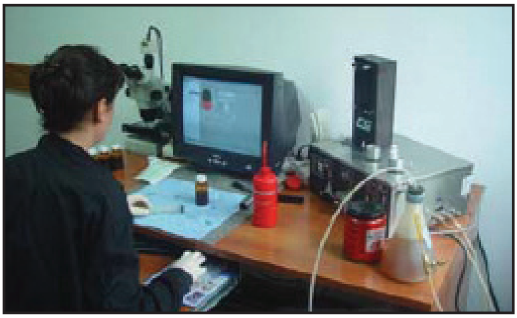
Rominserv is a Romanian company which integrates industrial services, maintenance, project management and technical upgrades. The company maintains a Predictive Maintenance Laboratory where the vibration and lube oil data is processed and evaluated in order to predict when a machine will need to receive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

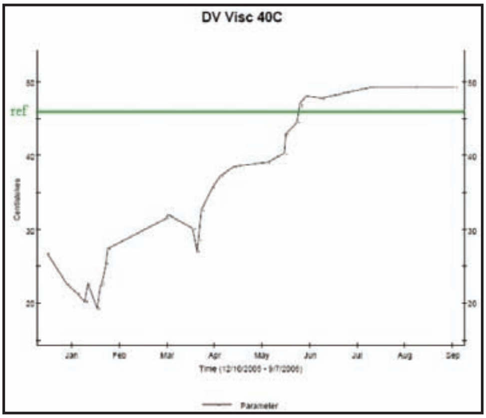
Such was the case when an early break of the safety bolt on the rotor of a critical pump was detected. The fault could have resulted in a sudden pump breakdown that would have shut down an entire unit. However, the problem was discovered in time, and the flow was shifted to another pump without incident. 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.

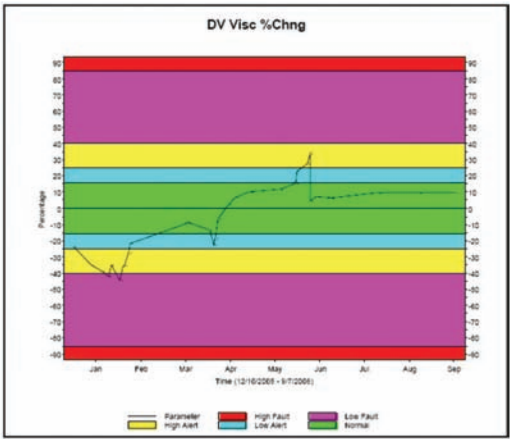
Romniserv Case in Point #2 - Lube Oil Results

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 admissable levels (mauve code). In addition, the water and particle quantity was within normal limits.

Figure 15-2: Trivector Plot

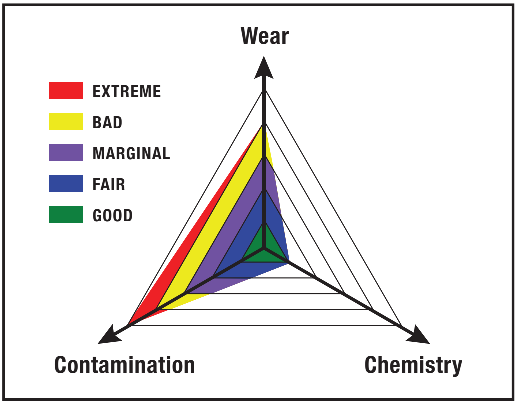


Table 1: Root Cause Failure Analysis

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"

16: Mining Case Study

In-House Oil Analysis

An operating gold mine has become a firm believer in the benefits of on-site, timely oil analysis to identify mechanical problems in loading and haulage units before they result in major repairs and unscheduled downtime. The low-grade surface mining operation previously 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 turnaround to ship samples and receive results created a risk that damage could occur before the results were obtained.

The mine overcame this problem by creating an in-house lab based on Spectro's Spectroil Q100 rotating disk electrode (RDE) atomic emission spectrometer and SpectroFTIR Q400 oil analyzer. It now can obtain oil analysis results in just 12 minutes and the reduced cost per sample has made it possible to increase the number of samples analyzed by almost tenfold. In its first year of operation, said Spectro, the in-house lab has helped the mine realize significant savings in downtime in repairs—savings that quickly paid for the purchase price of the instruments.

The mine's loading and haulage fleet includes a large excavating shovel powered by a 2,600-hp diesel, and 250-ton payload haul trucks powered by 16-cylinder, 2,500-hp diesels. These large items of equipment are worth approximately $15,000 per hour when operating, so minimizing downtime is critical to profitability. Repair costs for this equipment can be enormous; for example, the cost of rebuilding an engine is about $400,000. Even more important, the sudden failure of a major system on heavy equipment could create a safety issue.

Figure 16-1: Spectroil Q100 Spectrometer

In the past, the gold mine had performed some oil monitoring in-house with a very basic analysis tool, but its lack of reliability forced the mine to depend primarily on an outside lab—which provided reliable results but involved a process that took two days to ship samples 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 unplanned downtime and substantially higher repair costs. In addition, the outside lab charged about $12 per sample and the mine paid $15 per sample for shipping costs. These costs limited the mine to analyzing an average of five samples per day.

The mine's in-house lab was set-up to use 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. Management also plans to purchase a SpectroVISC Q300 semi-automatic kinematic viscometer and SpectroT2FM Q500 analytical ferrography laboratory in the near future.

The mine 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

Figure 16-2: SpectroT2FM Q500 Analytical Ferrography Laboratory

history of the machine and other similar machines to help determine the best course of action.

The in-house lab has already identified and helped prevent catastrophic damage from four major equipment problems, each of which would have cost more to fix than the entire cost of outfitting the in-house lab. In one case, the inhouse 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 explained.

“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. Re-bearing cost only about 12 hours of downtime compared to 88 hours if we had to change the engine.”

In another case, the lab detected copper levels in the transmission oil of a large wheel 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 save warranty costs. During a failed-engine overhaul, the rebuilder took a sample from the bottom of the pan and claimed to have found high levels of glycol—and then blamed the failure on a glycol leak, which would not have been covered by the warranty. The mine then sent similar samples to its in¬house lab and to an independent lab for verification—both of which came back with findings of “no glycol” in the sample. Consequently, the manufacturer covered the cost of rebuilding the engine, which saved the mine 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 mine’s RCM coordinator stated. “We have already saved four engines in 18 months of operating the inhouse 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 1,500 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.”

Unit Lets Non-expert Workers Check Oil Viscosity

Spectro recently introduced the latest evolution of its SpectroVisc Q3000 series of portable kinematic viscometers that are designed to quickly measure lubricant viscosity in the field. The range of the new SpectroVisc Q3050 model extends from 1cSt-680cSt at 40°C; the new functionality is the result of a new polished cell that provides the range and performance enhancements. The unit can also calculate 100°C viscosity values with the input of the VI index.

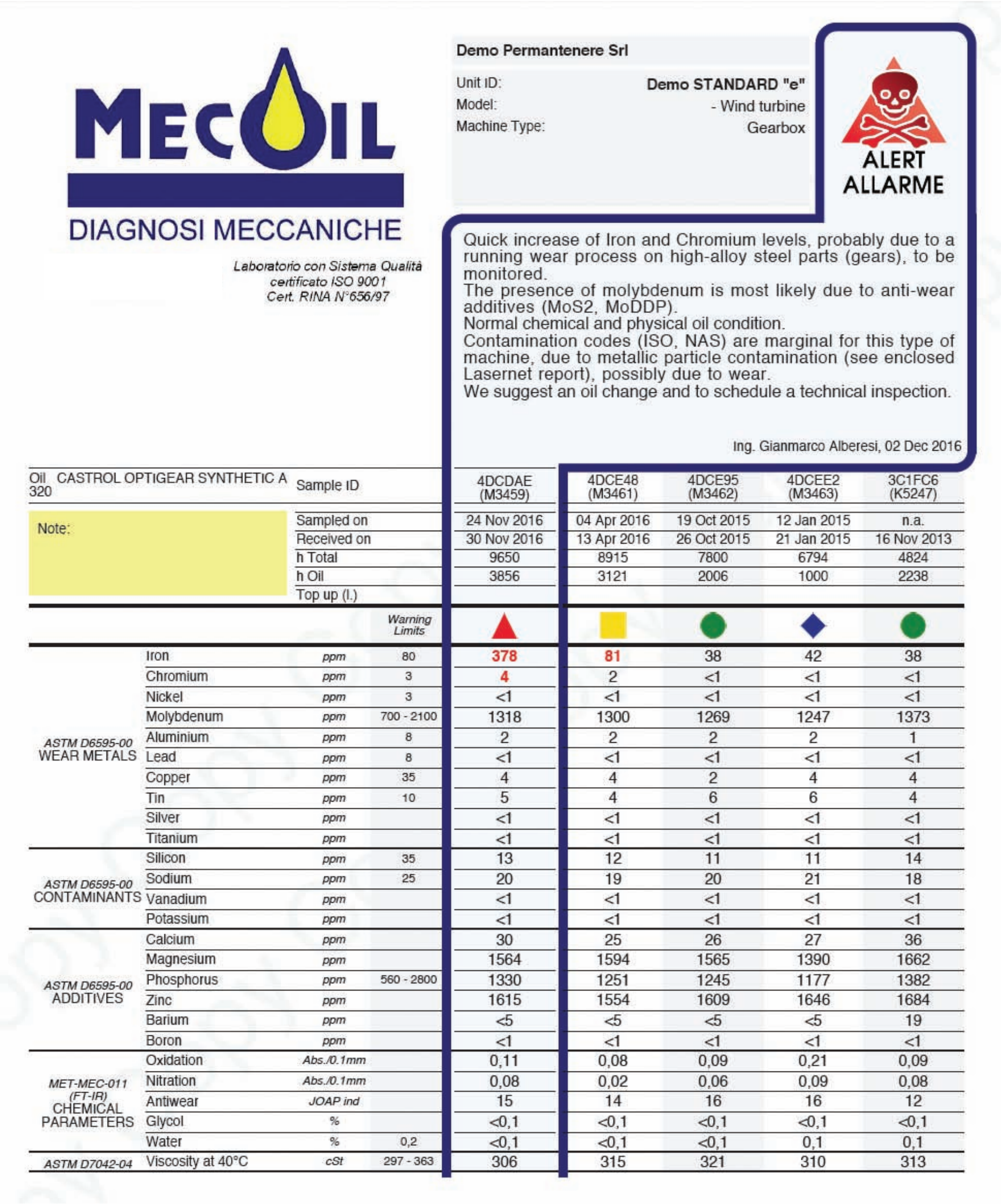
SpectroVisc Q3000 viscometers use a patent-pending split cell design that enables measurement of kinematic viscosity using only a few drops (60μL) of oil. In addition, no solvents are required for cleaning sampling cell surfaces. The compact units fit easily into a toolbox or carrying bag and operate more than six hours between recharges.

The extended measurement range of the SpectroVisc Q3050 kinematic viscometer will enable users to handle a wider range of testing applications and thereby increase their ability to control operational and maintenance costs, minimize downtime, and prevent catastrophic equipment failures. The SpectroVisc’s small size, zero solvent usage and minimal sample volume features are consistent with our mission of developing and deploying field portable, analytical tools for operation by nonexpert users.

Figure 16-3: SpectroVisc Q3050 Portable Viscometer

Appendix 1. A Detailed Laboratory Report

In this appendix, a detailed oil analysis report is shown as an example of different types of parameters tested by an oil analysis lab (Courtesy of Mecoil).



MECOIL
DIAGNOSI MECCANICHE

Laboratorio con Sistema Qualità
certificato ISO 9001
Cert. RINA N°656/97

Demo Permantenere Srl

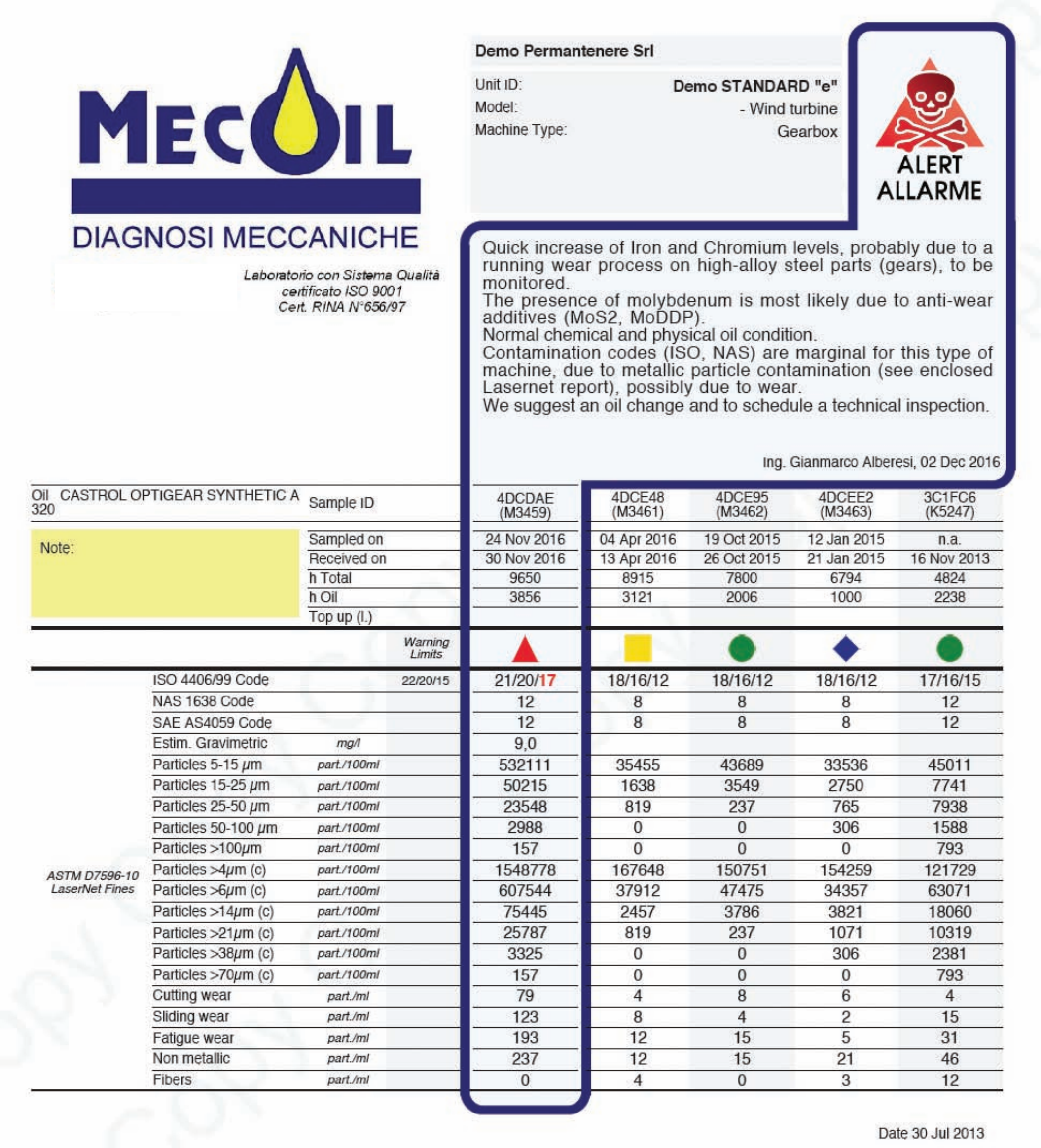
Unit ID:	**Demo STANDARD "e"**
Model:	- Wind turbine
Machine Type:	Gearbox

ALERT
ALLARME

Quick increase of Iron and Chromium levels, probably due to a running wear process on high-alloy steel parts (gears), to be monitored.
The presence of molybdenum is most likely due to anti-wear additives (MoS2, MoDDP).
Normal chemical and physical oil condition.
Contamination codes (ISO, NAS) are marginal for this type of machine, due to metallic particle contamination (see enclosed Lasernet report), possibly due to wear.
We suggest an oil change and to schedule a technical inspection.

Ing. Gianmarco Alberesi, 02 Dec 2016

Oil CASTROL OPTIGEAR SYNTHETIC A 320	Sample ID			4DCDAE (M3459)	4DCE48 (M3461)	4DCE95 (M3462)	4DCEE2 (M3463)	3C1FC6 (K5247)
Note:	Sampled on			24 Nov 2016	04 Apr 2016	19 Oct 2015	12 Jan 2015	n.a.
	Received on			30 Nov 2016	13 Apr 2016	26 Oct 2015	21 Jan 2015	16 Nov 2013
	h Total			9650	8915	7800	6794	4824
	h Oil			3856	3121	2006	1000	2238
	Top up (l.)							
			Warning Limits	▲	■	●	◆	●
ASTM D6595-00 WEAR METALS	Iron	*ppm*	80	378	81	38	42	38
	Chromium	*ppm*	3	4	2	<1	<1	<1
	Nickel	*ppm*	3	<1	<1	<1	<1	<1
	Molybdenum	*ppm*	700 - 2100	1318	1300	1269	1247	1373
	Aluminium	*ppm*	8	2	2	2	2	1
	Lead	*ppm*	8	<1	<1	<1	<1	<1
	Copper	*ppm*	35	4	4	2	4	4
	Tin	*ppm*	10	5	4	6	6	4
	Silver	*ppm*		<1	<1	<1	<1	<1
	Titanium	*ppm*		<1	<1	<1	<1	<1
ASTM D6595-00 CONTAMINANTS	Silicon	*ppm*	35	13	12	11	11	14
	Sodium	*ppm*	25	20	19	20	21	18
	Vanadium	*ppm*		<1	<1	<1	<1	<1
	Potassium	*ppm*		<1	<1	<1	<1	<1
ASTM D6595-00 ADDITIVES	Calcium	*ppm*		30	25	26	27	36
	Magnesium	*ppm*		1564	1594	1565	1390	1662
	Phosphorus	*ppm*	560 - 2800	1330	1251	1245	1177	1382
	Zinc	*ppm*		1615	1554	1609	1646	1684
	Barium	*ppm*		<5	<5	<5	<5	19
	Boron	*ppm*		<1	<1	<1	<1	<1
MET-MEC-011 (FT-IR) CHEMICAL PARAMETERS	Oxidation	*Abs./0.1mm*		0,11	0,08	0,09	0,21	0,09
	Nitration	*Abs./0.1mm*		0,08	0,02	0,06	0,09	0,08
	Antiwear	*JOAP ind*		15	14	16	16	12
	Glycol	*%*		<0,1	<0,1	<0,1	<0,1	<0,1
	Water	*%*	0,2	<0,1	<0,1	<0,1	0,1	0,1
ASTM D7042-04	Viscosity at 40°C	*cSt*	297 - 363	306	315	321	310	313



MECOIL
DIAGNOSI MECCANICHE

Laboratorio con Sistema Qualità certificato ISO 9001 Cert. RINA N°656/97

Demo Permantenere Srl

Unit ID:	**Demo STANDARD "e"**
Model:	- Wind turbine
Machine Type:	Gearbox

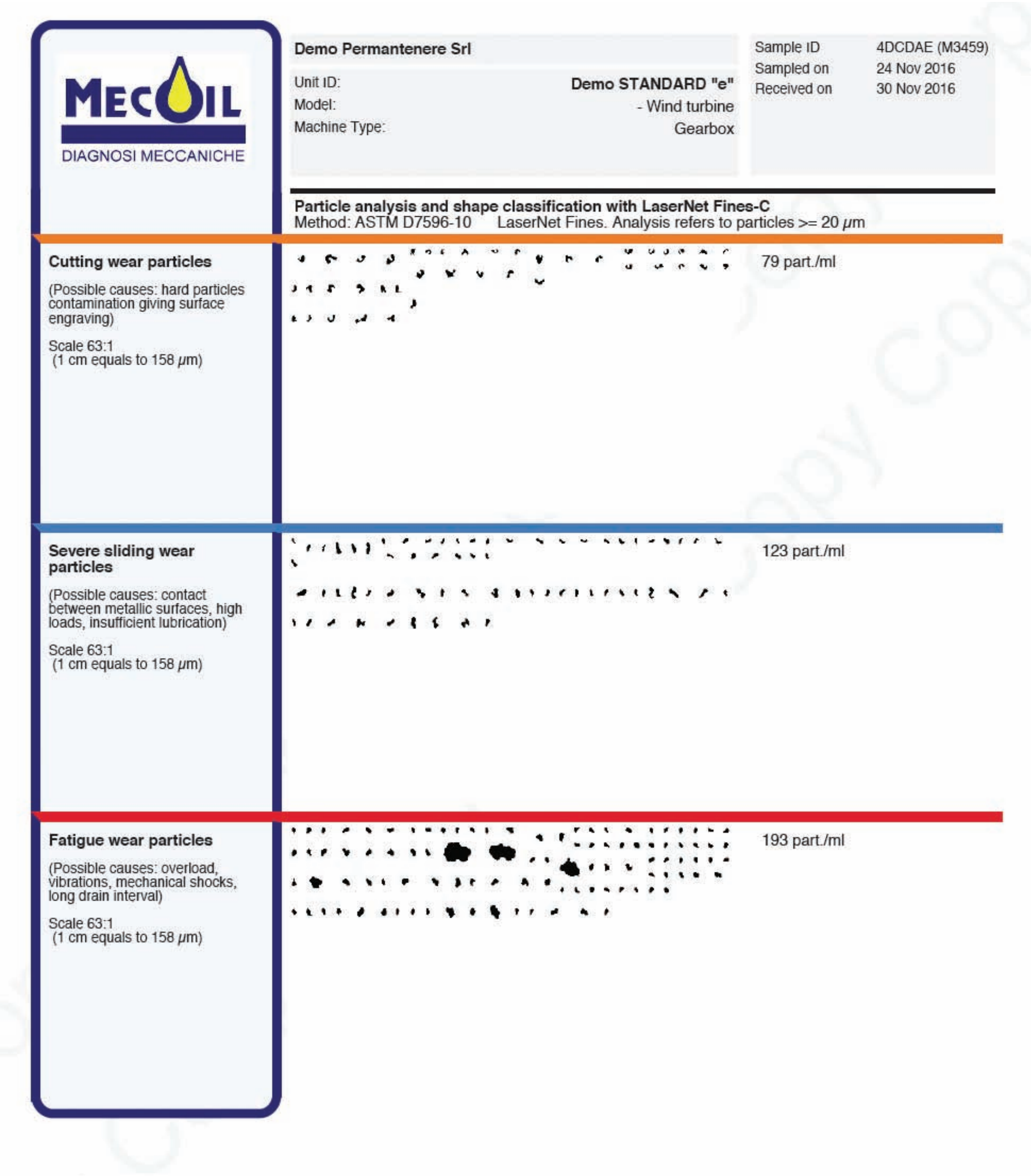
ALERT
ALLARME

Quick increase of Iron and Chromium levels, probably due to a running wear process on high-alloy steel parts (gears), to be monitored.
The presence of molybdenum is most likely due to anti-wear additives (MoS2, MoDDP).
Normal chemical and physical oil condition.
Contamination codes (ISO, NAS) are marginal for this type of machine, due to metallic particle contamination (see enclosed Lasernet report), possibly due to wear.
We suggest an oil change and to schedule a technical inspection.

Ing. Gianmarco Alberesi, 02 Dec 2016

Oil CASTROL OPTIGEAR SYNTHETIC A 320	Sample ID			4DCDAE (M3459)	4DCE48 (M3461)	4DCE95 (M3462)	4DCEE2 (M3463)	3C1FC6 (K5247)
Note:	Sampled on			24 Nov 2016	04 Apr 2016	19 Oct 2015	12 Jan 2015	n.a.
	Received on			30 Nov 2016	13 Apr 2016	26 Oct 2015	21 Jan 2015	16 Nov 2013
	h Total			9650	8915	7800	6794	4824
	h Oil			3856	3121	2006	1000	2238
	Top up (l.)							
			Warning Limits	▲	■	●	◆	●
	ISO 4406/99 Code		22/20/15	21/20/17	18/16/12	18/16/12	18/16/12	17/16/15
	NAS 1638 Code			12	8	8	8	12
	SAE AS4059 Code			12	8	8	8	12
	Estim. Gravimetric	*mg/l*		9,0				
	Particles 5-15 µm	*part./100ml*		532111	35455	43689	33536	45011
	Particles 15-25 µm	*part./100ml*		50215	1638	3549	2750	7741
	Particles 25-50 µm	*part./100ml*		23548	819	237	765	7938
	Particles 50-100 µm	*part./100ml*		2988	0	0	306	1588
	Particles >100µm	*part./100ml*		157	0	0	0	793
ASTM D7596-10 LaserNet Fines	Particles >4µm (c)	*part./100ml*		1548778	167648	150751	154259	121729
	Particles >6µm (c)	*part./100ml*		607544	37912	47475	34357	63071
	Particles >14µm (c)	*part./100ml*		75445	2457	3786	3821	18060
	Particles >21µm (c)	*part./100ml*		25787	819	237	1071	10319
	Particles >38µm (c)	*part./100ml*		3325	0	0	306	2381
	Particles >70µm (c)	*part./100ml*		157	0	0	0	793
	Cutting wear	*part./ml*		79	4	8	6	4
	Sliding wear	*part./ml*		123	8	4	2	15
	Fatigue wear	*part./ml*		193	12	15	5	31
	Non metallic	*part./ml*		237	12	15	21	46
	Fibers	*part./ml*		0	4	0	3	12

Date 30 Jul 2013



MECOIL
DIAGNOSI MECCANICHE

Demo Permantenere Srl

Unit ID:	**Demo STANDARD "e"**
Model:	- Wind turbine
Machine Type:	Gearbox

Sample ID	4DCDAE (M3459)
Sampled on	24 Nov 2016
Received on	30 Nov 2016

Particle analysis and shape classification with LaserNet Fines-C
Method: ASTM D7596-10 LaserNet Fines. Analysis refers to particles >= 20 μm

Cutting wear particles

(Possible causes: hard particles contamination giving surface engraving)

Scale 63:1
(1 cm equals to 158 μm)

79 part./ml

Severe sliding wear particles

(Possible causes: contact between metallic surfaces, high loads, insufficient lubrication)

Scale 63:1
(1 cm equals to 158 μm)

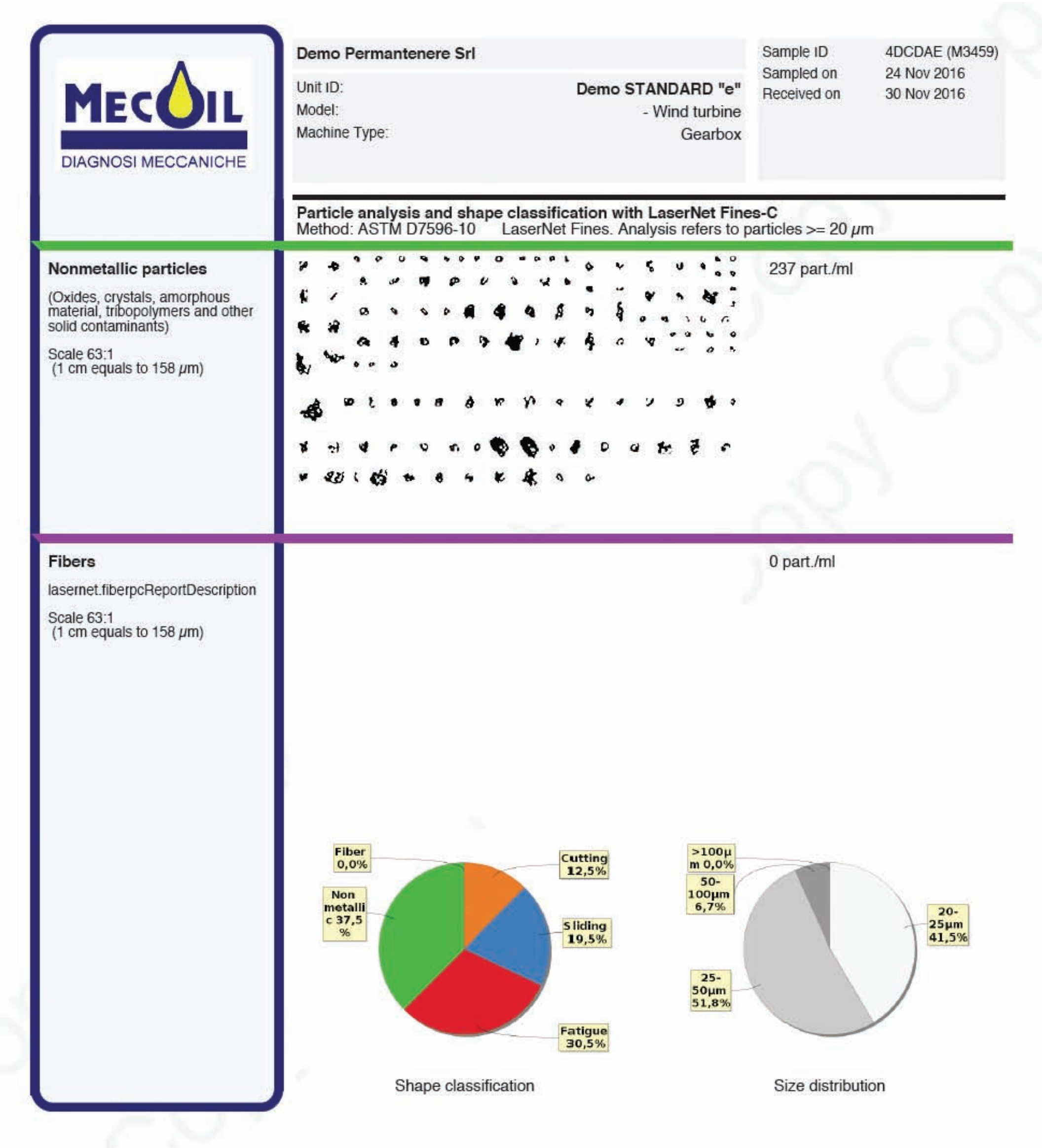
123 part./ml

Fatigue wear particles

(Possible causes: overload, vibrations, mechanical shocks, long drain interval)

Scale 63:1
(1 cm equals to 158 μm)

193 part./ml



MECOIL
DIAGNOSI MECCANICHE

Demo Permantenere Srl

Unit ID:	**Demo STANDARD "e"**
Model:	- Wind turbine
Machine Type:	Gearbox

Sample ID	4DCDAE (M3459)
Sampled on	24 Nov 2016
Received on	30 Nov 2016

Particle analysis and shape classification with LaserNet Fines-C
Method: ASTM D7596-10 LaserNet Fines. Analysis refers to particles >= 20 µm

Nonmetallic particles

(Oxides, crystals, amorphous material, tribopolymers and other solid contaminants)

Scale 63:1
(1 cm equals to 158 µm)

237 part./ml

Fibers

lasernet.fiberpcReportDescription

Scale 63:1
(1 cm equals to 158 µm)

0 part./ml

Shape classification

Size distribution