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Remote, challenging operations? No problem for this advanced technology. It pays off in real time by going directly to your equipment, anywhere in the world, any time you need it.

Lubricating fluids degrade over time depending on various external and internal influences, including type and age of equipment, ambient temperature and humidity and degree of use and load on equipment, etc. It is well established that monitoring the health of lubricating fluids is an important and necessary part of high-value machinery maintenance. The traditional approach for determining the condition of these vital lubricants is to take a sample, send it off for analysis at a commercial testing lab, then track trends in changes in key lube parameters over time. When these analyses indicate a problem, corrective actions such as refreshing or changing the lubricant are taken.

Hydroelectric power generating operations are just one place where IR spectroscopy of lubricants can pay off beautifully.

As companies move from preventive maintenance to proactive maintenance, there is increasing interest in onsite lubricant testing because results can be obtained much faster—and they may be more trustworthy. It allows lubrication specialists and maintenance personnel to take decisive action right away. This latter point is important since some of the degradation processes in lubricants occur nonlinearly in time and more quickly than one might expect, which can lead to increased equipment wear or failure. Of course, the ability to use on-site testing equipment is predicated on the ability of the testing equipment manufacturers to make their products straightforward to use and provide valuable information.

A number of analysis methods have made the jump from use by experts off site to routine use by lubrication specialists on site. One technique not making that jump—until now—has been infrared spectroscopy. Infrared has been used for years to evaluate lubricating fluids, but virtually always in off-site commercial labs. Now, though, infrared analysis also is available for use in on-site facilities.

Monitoring critical lubricant parameters

There are several key parameters for which infrared is capable of providing highly accurate information in lubricants including:

- The level of water present
- The amount of oxidation and nitration by-products
- The amount of anti-wear, anti-oxidation and extreme pressure additives remaining

All of these parameters are critical—*and some can be measured with other methods*. No other technology, however, can provide information on all parameters simultaneously, in less than two minutes. The use of infrared analysis for each parameter will be explored here.

Infrared analysis for water

The amount of water that is present in lubricants is critical to the performance and longevity of the lubricated equipment. Lubricant properties affected by the presence of water include viscosity (measure of the oil's resistance to flow), specific gravity (density of the oil relative to that of water), and the surface tension (a measure of the stickiness between surface molecules of a liquid). All of these properties are important for the ability of the oil to coat, lubricate and protect the critical mechanical clearances. In addition, the presence of water can accelerate additive depletion and contribute to chemical degradation mechanisms such as oxidation, nitration and varnish formation.

The ability to measure water on-site provides a substantial benefit to ensure accuracy of results. Off-site analysis for trace water may be compromised due to variability of water concentration introduced by storage, transportation or shipment of a sample. Furthermore, some lubricants contain de-emulsifying additives that cause microscopic water droplets to separate concentrate in layers at the bottom and sides of sampling containers. This de-emulsifying action takes time to occur and can cause large variations in analytical measurements. Furthermore, lubricant samples can lose water due to evaporation and loss to the sample container walls. To obtain an accurate picture of the amount of water present, measurement should be made soon after the sample is pulled from the machine.

Analytical determination of water in lubricants typically is carried out using the well-established Karl Fischer (KF) coulometric titration. KF has some practical drawbacks for on-site analysis including complicated sample preparation, the use of hazardous and expensive chemical reagents and length of time required to perform the analysis. With these issues in mind, KF analysis is still considered the "gold standard" method for analyzing water in oil because it provides accurate and precise answers. Under ideal conditions, Karl Fischer has an accuracy of 3-5% for prediction of water in lubricants.

While infrared spectroscopy provides an easy means to measure water, only recently has this technology been able to provide the accuracy and range desired by the lubrication industry. New developments in the ability to use FTIR spectroscopy to carry out customized methods have now made the analysis of low levels of water in lubrication possible, which overcomes earlier technical difficulties. These new methods, coupled with a dedicated on-site infrared analyzer, measure the concentration of water in mineral-based oils with an accuracy and range equivalent to the Karl Fischer method. FTIR allows this measurement to be carried out on a single drop of lubricant, requiring no hazardous or expensive reagents, and it takes significantly less time to complete than KF.

Methods to directly measure water in mineral oils via infrared spectroscopy have been available for over 30 years. For example, the ASTM 2412E method was originally designed for use with motor oil. Routinely containing 1000 to 2000 ppm of water, motor oil has additives that solvate the water into the oil. The methods developed to measure water in these oils by infrared analysis were targeted at large concentration and had correspondingly large errors associated with them. Other lubricants (such as turbine oil) solvate significantly less water—*typically it's 50 to 100 ppm*. In these lubricants, higher levels of water form small droplets that eventually settle to the bottom of the turbine oil. If the ASTM 21412 method for water is used for turbine oil, measurement variability of up to 40% on replicate samples is observed.

The primary reason the conventional method for measuring water in oil by FTIR produces a high error in turbine oils is water separation—*water separates into small droplets in turbine oil*. These small droplets scatter instead of absorb infrared light, and only the light that is absorbed contributes to the measurement of water. Over time, it became clear that a means of stabilizing the water in the oil would be needed to reduce variability.

Table I. Water Measured by FTIR Infrared System vs. Karl Fischer Titration

PAL (ppm)	KF (ppm)	Difference (ppm)	% Error
508	504	4	0.8
1054	965	89	9.2
2043	2002	41	2.0
2946	2838	108	3.8
4710	4753	43	0.9

Water stabilization method for infrared analysis

A new method (patent pending) has been developed for the measurement of water in turbine oil. This method, reflected by the data in Table I, uses a surfactant to distribute and stabilize the water in the oil, creating a stable emulsion with uniform water droplet size. Addition of approximately 3% of a premixed non-ionic polyethylene oxide based surfactant blend and gentle mixing effectively stabilizes the water in the lubricant.

Determining degree of oxidation and antioxidant depletion

Oxidation is the most significant cause of lubrication breakdown. It occurs when the hydrocarbon components of the lube combine with oxygen to form a wide range of harmful by-products including ketones, aldehydes and carboxylic acids. Once these compounds form, they in turn combine with other species in the lube and form even more unwanted degradative products. Virtually all of the chemical species that result from oxidative processes can be detected and measured by infrared analysis (Fig. 1). Early detection of these species allows for remediation action to slow down the oxidation process.

The phenolic and aminic antioxidants in lubricants function as preservatives that prevent the oil from oxidizing. Oxidation causes lubricants to quickly lose viscosity and the wetting characteristics that protect metal contact surfaces and prevent wear. Oxidation arises from a combination of sources—*including elevated temperatures, extreme pressures, high shear conditions and the presence of water and metal particles*—and is accelerated by electrostatic sparking, particularly in certain gas turbine systems. Although antioxidants inhibit the formation of these decomposition products, once the antioxidants are consumed, oxidation accelerates exponentially and at a certain critical point corrective action has negligible benefit. On-site analysis offers a significant benefit in this regard by ensuring that both the antioxidant levels and the amount of oxidation present are known in time for corrective action to be taken before the critical point is reached.

Infrared compared to other oxidation-measuring technology
Infrared analyzers require a drop of neat oil—with no sample preparation. Voltammetric systems require careful pipetting techniques and an extraction step involving an electrolyte solution. The extraction step used in voltammetric systems assumes that all of the antioxidants are extracted from the oil into the electrolyte solution. However, extraction efficiencies are variable for additives in oils. Ranging from 50-90%, these efficiencies may result in 10-50% of additives being left in the oil after extraction, and thus not being measured. Moreover, voltammetric electrodes require maintenance, such as conditioning in buffer solutions. Metal particles, water or organic salts (i.e. ionized carboxyls such as copper carboxylates) will not interfere with the antioxidant measurements using infrared spectroscopy.

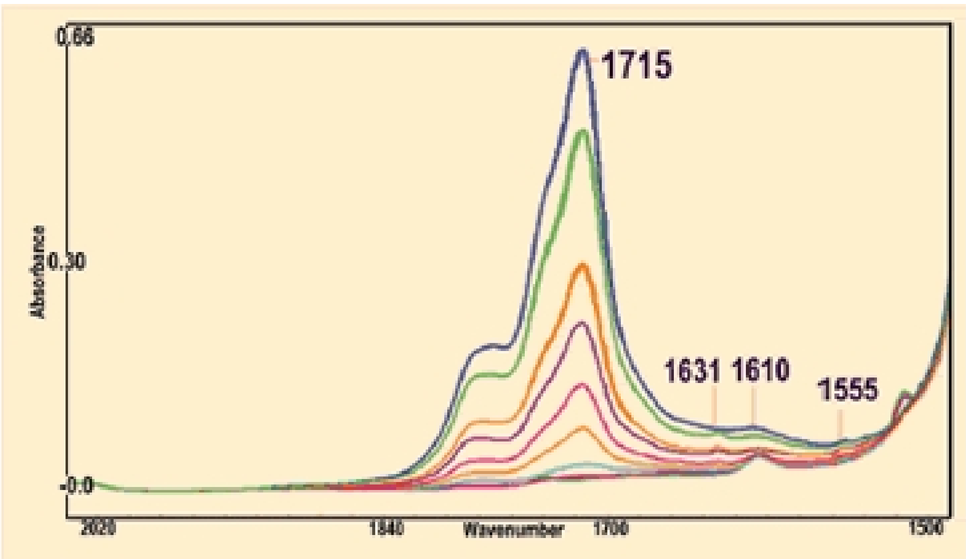

Fig. 1. FTIR detects the formation of carboxylic acids and increase in the peak at 1715cm-1 and nitration as an increase in the 1631 peak. Carboxylate sales are seen at 1555cm-1. In short, all the increases in the peaks in the region 1800 to 1500 cm-1 are caused by nitration.

Conclusion
Real-time, on-site FTIR analysis offers a number of potential—*and important*—benefits to lubrication specialists and maintenance personnel. They include the ability to:

- Analyze lubricants more frequently, especially when previous analyses indicate that machinery needs more careful monitoring… When the performance of lubricating fluid begins to degrade, or if earlier analyses indicate the presence of a mechanical problem, it is important to monitor the lubricant more frequently because the process of deleterious change can accelerate rapidly.

- Help reduce machinery wear caused by rapid oil breakdown and to detect problems that could cause catastrophic failures… For example, an anti-freeze leak causes excessive levels of water and glycol to be present in engine oil; these levels can be readily detected by FTIR. More frequent monitoring of engine oil by real-time FTIR can quickly catch these contaminants before they have a chance to cause catastrophic damage to an engine.
- Ascertain the condition of lubricants in remotely deployed equipment, for which the delay in receiving information from off-site labs may be unacceptable… On-site FTIR analysis minimizes the need to send lubrication samples to off-site labs for condition-based monitoring. It is especially important that equipment operating in these remote locations be carefully monitored since ambient conditions may be particularly challenging.
- Act as the supporting analytical technology in programs designed to bring lubricants back to spec via readditization… FTIR is a powerful method for analysis of anti-wear and anti-oxidation additives. More companies are looking to extend the use of lubricants by refreshing critical additives to bring the lubricant back to spec. Real-time, on-site FTIR can be a powerful tool for determining how much additive should be recharged and for monitoring the overall refreshed oil composition.
- Enable maintenance personnel to make better decisions on when to send oil samples for full analysis… Real-time FTIR is an excellent screening technology to detect problems with both the lubricating fluid and the lubricated equipment. More frequent screening with FTIR enables personnel to make informed decisions on when to send samples for full elemental analysis, in order to try to pinpoint specific internal machine problems that may indicate excessive mechanical wear.
- Determine that incoming lubricants are properly formulated, not contaminated in shipping or mislabeled, and that the correct lubrication fluid is charged into the machinery… It is vitally important to use lubricants that meet the equipment manufacturer's specifications. When special lubricants are ordered and shipped, mistakes can occur in formulation or in delivery. A portable FTIR system can be used right at the loading dock or at the tanker truck delivering fluids, to ensure that the delivery matches the expected formulation.

Infrared spectroscopy provides an immediate snapshot of the overall health of lubricating fluids—*it is a window to the vital signs of both the lubricants and the equipment that lubricants protect.* Few analytical techniques provide so much information about key parameters that affect lubricating fluid life and engine health. With the new generation of infrared analyzers, the technology can now be used where it is needed, either on site or at site—wherever machinery is in use. That includes some of the most remote and challenging industrial operations on earth. This new approach assists maintenance, service and equipment reliability personnel in making rapid, actionable decisions based on objective analytical data.
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