Raising the bar in FT-IR

getting the most out of on-site oil condition monitoring

Introduction

Lubrication is a vital component of machinery and, as with any component, early detection of wear is essential for timely replacement, avoiding unnecessary maintenance while reducing the risk of catastrophic breakdowns. Oil inevitably degrades with use and is exposed to various sources of contamination, both internal and external, which can lead to its degradation and reduced effectiveness in protecting vital components. Early identification of impending lubricant failure can prevent costly damage.

Infrared (IR) analysis is a rapid, inexpensive test that can provide specific information about the chemical condition of the oil, also allowing inferences about the state of the machine component from which the sample was taken. Trend data can be gathered by regular sampling to monitor engine performance and operating conditions. This allows the oil to be changed at optimal intervals, which has two major benefits. Costs are reduced by carrying out the oil change only when necessary and problems are identified early, which allows the appropriate preventative steps to be taken.

The benefits of oil condition monitoring can be further increased by complementing routine measurements at a centralised laboratory with more rapid on-site analysis using a portable analyser. This article reviews infrared oil condition monitoring and describes the design considerations for an on-site oil analyser, the In-Service Lubricants FT-IR Analysis System from PerkinElmer.

Infrared spectroscopy for oil condition monitoring

The infrared (IR) spectrum is obtained by measuring the transmission of infrared light through a sample of the oil. Molecular species present in the oil absorb light at characteristic frequencies as vibrational modes within the molecules are excited. IR spectroscopy has a number of properties that make it a valuable tool in oil condition monitoring. The intensity of an absorption band is proportional to the concentration of the absorbing species, so IR measurements provide information that is both specific and quantitative. One of the key advantages of IR spectroscopy is the rapidity of the measurement: minimal sample preparation is required (apart from homogenisation and filtering) and the spectrum can be obtained within a few seconds.

IR measurements are sensitive to a number of additives, degradation products and potential contaminants that can be found in oil. These include:

Contamination

- **Soot** is a by-product of incomplete combustion and, while the oil may tolerate a significant concentration of soot, a greater-than-expected increase over time may indicate an incorrect fuel to air ratio, clogged air filters or an over-extended oil change period.
- The presence of **water** and **glycol** in the oil indicates a leak from the cooling system and requires urgent attention. Water alone may be due to condensation at low operating temperatures. Water in hydraulic systems may be due to condensation in oil reservoirs.
- **Unburned fuel** in the oil may indicate poor combustion. While fuel may be chemically rather similar to the lubricant base oil, the aromatic content of the fuel can allow a calibration to be developed.

Chemical degradation

- **Oxidation** occurs when oil is exposed to oxygen at elevated temperatures. The reaction pathways are very complex but the end results are thickening, varnish formation and a build-up of carboxylic acids that increase the acidity of the oil and can lead to corrosion. Oil oxidation occurs slowly as sacrificial antioxidant additives are used up and then rapidly once the additives have been depleted. Regular monitoring of the oil is important to allow corrective action to be taken before this sudden increase in oxidation occurs.
- **Nitric oxides** are produced during combustion, and these can also oxidise the oil, producing compounds containing nitrogen and oxygen. As with oxidation, this leads to thickening, varnish build-up and depletion of the oil base reserve. High levels of nitro-oxidation can indicate a number of faults, such as incorrect fuel to air ratio, incorrect spark timing, excessive loads, low operating temperatures or piston-ring blow by.
- Acidic **sulphur** compounds can be produced during combustion if sulphur compounds are present as impurities or additives in the fuel or lubricant. Again, this leads to the depletion of basic additives and eventually to corrosion.
- Synthetic oils with a polyol ester base are susceptible to hydrolysis at elevated temperatures if water is present. The **ester breakdown** products also contribute to the acidity of the oil and can sometimes form crystals that clog filters.

Additive depletion

There are numerous compounds added to oils to confer desirable functional properties. These additives can be depleted during use, and monitoring this depletion can provide an early warning of impending lubricant failure.

- *Anti-wear* additives such as zinc di-alkyl or di-aryl dithiophosphates (ZDDPs) prevent direct metal-to-metal contact by forming a coating on metal surfaces activated by frictional heat. These additives can be depleted by hydrolysis or oxidation, and this can result in an increase in wear rate.
- *Phenolic antioxidant* compounds are often used in turbine lubricants. Depletion of the antioxidant is followed by a rapid increase in the rate of oxidation.

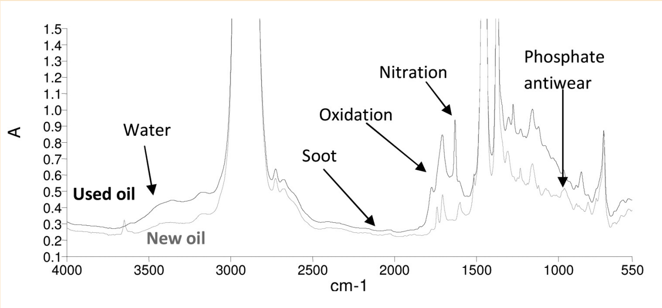
Figure 1 shows example spectra of a new oil and the same oil after an extended period of use. The chemical changes are dramatic and are easily measured with a high quality FT-IR

Continued on page 18

Continued from page 16

system. Characteristic absorption features corresponding to some of the properties listed above are shown.

Figure 1



IR spectra of new and used lubricating oils, with the key diagnostic absorption bands labelled.

Standardised test methods

The Joint Oil Analysis Programme (JOAP) run by the US military developed infrared test methods for lubricants, and these have become widely used. More recently, ASTM International introduced a series of standard practices and test methods (see ASTM E2412, D7412, D7414, D7415, D7418). These methods are all supported by recent software such as PerkinElmer's OilExpress.

IR spectroscopy allows quantitative calibrations to be developed to allow accurate concentration measurements of important species in the oil. However, in practice this is unnecessary: in most cases it is sufficient to express results (and warning/alarm limits) in terms of the intensity of characteristic infrared absorptions. There are two fundamentally different approaches to the measurement. In the past, the most common method involved subtracting the spectrum of the unused oil from the spectrum of the used oil sample. Any features present in the difference must be due to changes that have occurred, such as degradation, additive depletion or contamination. This method simplifies the interpretation, but relies on having a valid oil reference spectrum for subtraction – something that is not always possible when, for example, the oil has been topped up with a different product.

At present, 'direct-trending' methods such as the JOAP and ASTM methods mentioned above are more widely used. These methods simply make measurements directly on the used-oil spectrum, and the interpretation of the results is made in the context of trending over the life of the oil. Condemnation limits can be established by examining trending results from numerous pieces of machinery. Since there is no need to calibrate each instrument, new systems can be deployed easily. However, this does require that the spectrometers used are capable of delivering repeatable and reproducible results.

Analysis in action: requirements for on-site measurements

The speed of IR measurement and the value and diversity of the results it provides have led to it becoming a routine method for oil analysis labs. Typically these labs operate with extremely high throughput and rely on FT-IR systems with automated sampling to achieve the required speed of analysis. The data presented in Figure 1 were obtained with a PerkinElmer OilExpress system, which supports ASTM, JOAP and user-defined analysis protocols and uses a powerful syringe-pump-based liquid autosampler to achieve throughput of over 50 samples/hour (for oils with viscosity of 200 cSt or below).

While high-performance laboratory FT-IR systems are mature and robust, there can be considerable delays involved in getting the sample from the machine to the test lab, particularly where the site is remote and in rugged terrain: precisely the environment where avoiding equipment breakdowns and down-time is most critical. This has led to increasing interest recently in taking oil condition measurement out of the lab and analysing samples at the point of collection. On-site measurements provide immediate feedback about the state of the oil: if the measurement indicates a severe fault, operation can be halted and the cause investigated before further damage occurs.

In the past, FT-IR oil condition monitoring systems have not been well suited to on-site analysis, suffering either from a lack of robustness, poor portability or complex user interfaces requiring expert operation. Modern instrumentation, however, makes it possible to obtain lab-quality results with a portable system.

Ruggedness and transportability

The recently launched Spectrum Two FT-IR spectrometer has been designed to meet the challenges of on-site analysis without compromising on performance. The instrument is compact (weighing just 13 kg) and easily transportable with the dedicated carry-case. With a sample scan taking less than 30 seconds, the system brings lab quality results to the field, providing confidence in the data collected.

The system can be operated from a bench in the site office, from a mobile lab or even a car boot. In case of an intermittent or unreliable power supply, the spectrometer can be powered from a battery pack for 4 hours or from a vehicle power socket.

Traditionally, FT-IR instruments have been confined to air-conditioned environments and have required frequent dessicant replacements because of the susceptibility of infrared spectrometer optics to damage by water vapour. The patented OpticsGuard technology in Spectrum Two effectively eliminates the ingress of water vapour into the instrument, enormously prolonging dessicant lifetime. The instrument can thus be used without concern even in very humid environments.

Analytical performance

The performance target for an on-site FT-IR system must be to obtain results that are indistinguishable from the laboratory-based system. If this condition is reached, operators can have far greater confidence in basing decisions on the analytical results without waiting for the central laboratory to receive the samples. To meet this requirement, it is important that the sampling method (such as a transmission flow-cell) and software algorithms are equivalent and that the instrument resolution and signal-to-noise performance are at a similar level.

The Spectrum Two In-Service Lubricants analysis system is based on a compact, high-performance FT-IR spectrometer, and

Continued on page 20

Continued from page 18

employs the same zinc selenide flow cell as is used by OilExpress. Consequently, the spectra and oil condition parameters measured on the smaller, transportable instrument are indistinguishable from those measured on the high-end automated system, as shown in Figure 2 and Table 1.

Figure 2

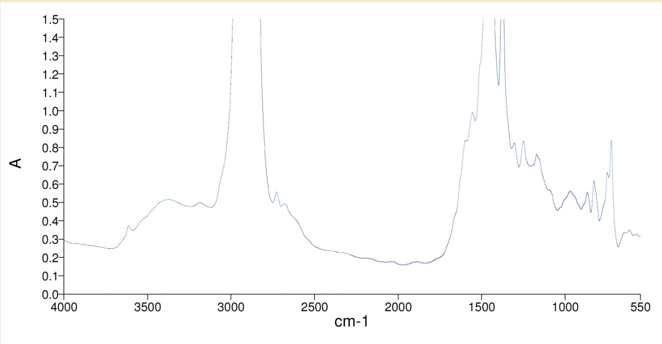


Figure 2. Spectra of the same diesel engine oil sample measured on an OilExpress system (black trace) and a Spectrum Two In-Service Lubricants Analysis System (blue trace). The corresponding oil condition parameters are shown in Table 1.

Table 1

Sample Name	Water	Soot	Oxidation	Nitration	Sulfate	Antiwear
OilExpress	91.3	16.6	6.65	22.4	27	17
Spectrum Two	91.5	16.2	6.59	22.4	26.9	16.8
Difference (%)	0.3	2.7	0.9	0.0	0.3	1.1

Table 1. Oil condition parameters for the spectra shown in Figure Y (obtained with the JOAP methodology).

This equivalence was further tested by measuring a number of samples obtained from an oil analysis laboratory on the Spectrum Two system and comparing the results to the original OilExpress data reported by the lab. For all the samples, excellent agreement was obtained, as shown in Figure 3.

Figure 3

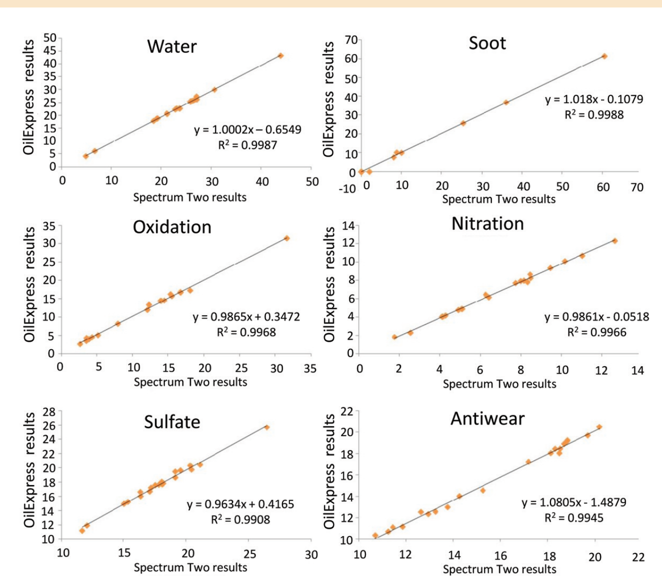


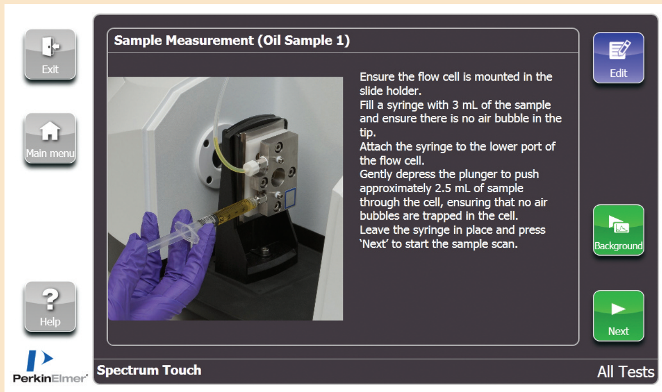
Figure 3. Comparison of oil condition parameters measured with an OilExpress system and a Spectrum Two system at a different site. Excellent correlations were obtained with the slope near unity and the intercept near zero in each case.

Usability

While equivalence of the analysis results is paramount, the user interface of an on-site analysis system has very different requirements from that of a laboratory instrument. It is critical that the system be easy to use for everyone, not just highly trained laboratory staff. However, it is also important that the software can be customised by the analysis manager with site-specific instructions, calibrations and alarm limits.

Spectrum Two addresses this with Spectrum Touch software, taking advantage of modern touch-screen hardware to present sampling instructions and standard operating procedures within a very intuitive, results-oriented interface. Out of the box, the system supports all current analysis methods provided by OilExpress. The method parameters are customisable, and site SOPs can easily be integrated into the interface.

Spectrum Touch



Integration with data systems

Results handling and reporting is an increasingly important area with the widespread use of laboratory information systems (LIMS). Spectrum Touch allows analysis reports to be displayed and printed, and stores a record of all analysed samples in a plain-text format that can easily be read by LIMS software. If a network connection is available, the results can even be sent automatically be e-mail, enabling expert interpretation at the oil analysis laboratory.

Conclusions

The value of oil condition monitoring programmes is now clearly recognised, and infrared spectroscopy has a key role to play as a tool for the rapid, sensitive measurement of oil degradation, contamination and additive depletion. Recent advances in technology mean it is now feasible to complement laboratory oil measurements with on-site analysis using a portable instrument. The In-Service Lubricants FT-IR Analysis System from PerkinElmer provides the ruggedness and transportability needed for on-site measurements while producing results that are equivalent to the market-leading OilExpress system. The intuitive operation of the system and easy integration with network data systems allow companies to maximise the benefits of their oil analysis programmes.

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